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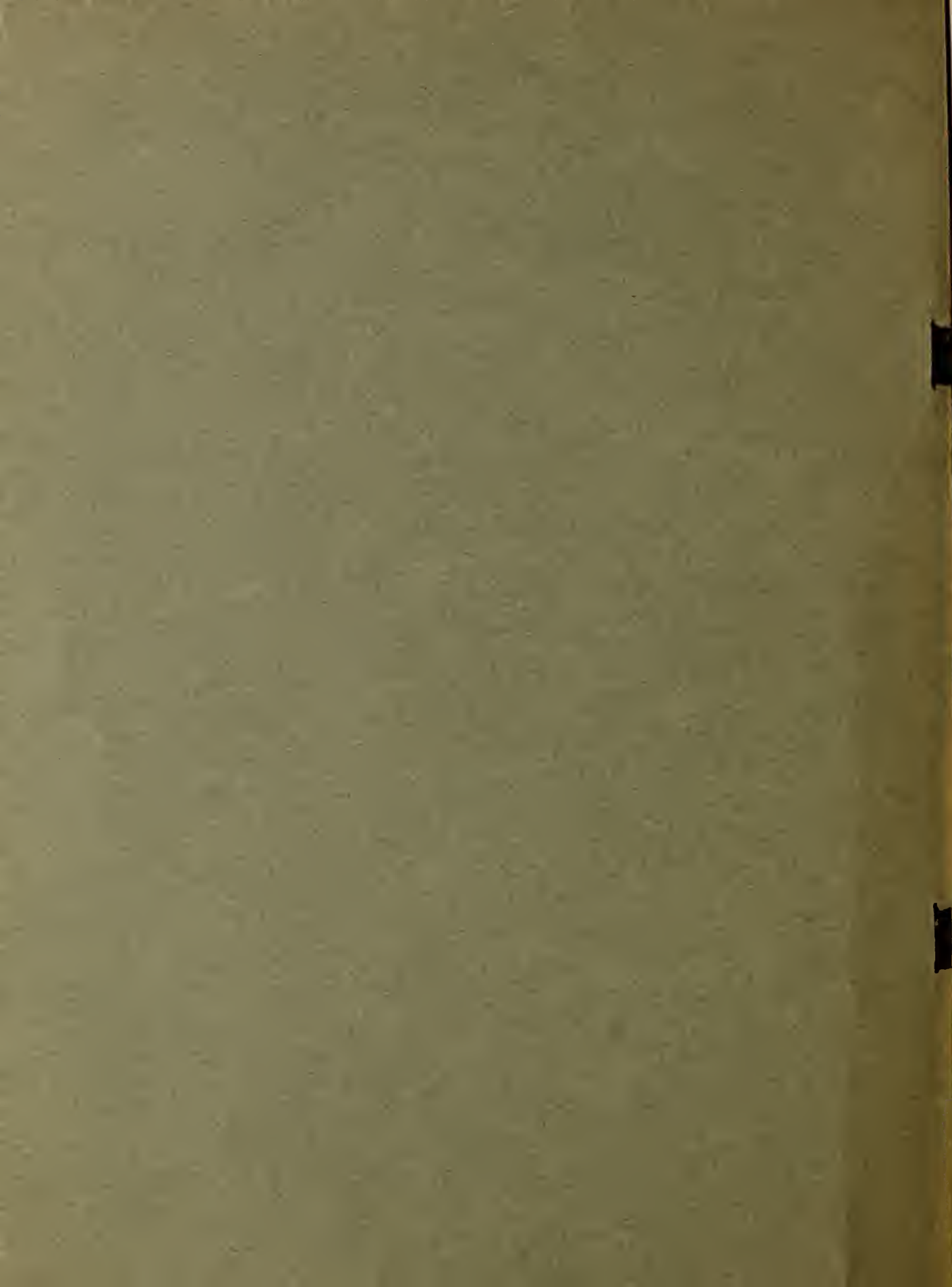
ABSTRACTS OF FOREIGN AND DOMESTIC PATENTS RELATING TO
DERRIS, LONCHOCARPUS, TEPHROSIA, AND ROTENONE

By

R. C. ROARK

Washington, D. C.

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United States Department of Agriculture
Bureau of Entomology and Plant Quarantine

ABSTRACTS OF FOREIGN AND DOMESTIC PATENTS RELATING TO

DERRIS, LONCHOCARPUS, TEPHROSIA, AND ROTENONE

(From the time the first application for a patent on derris
was filed (February 8, 1911) to April 1938

By R. C. Roark, Division of Insecticide Investigations

Eleven countries have issued patents which cover derris, cube, tephrosia, or rotenone products as insecticides, methods of extracting rotenone from plants, or processes of making dihydrorotenone or other derivatives.

The number of patents granted by each country is shown in the following list:

<u>Country</u>	<u>Number of patents</u>
Australia	3
Canada	14
France	6
Germany	7
Great Britain	23
Japan	3
Johore	1
Netherlands	1
Sweden	2
Switzerland	1
United States of America	42
Total	<u>110</u>

Australian Patents:

McDougall and Howles (59), in Australian patent 18,227/24, issued November 10, 1924, applied for June 16, 1924, make identical claims to the invention described in British patent 226,250.

The Zaidan Hojin Rikagaku Koukyujo (110), in Australian patent 15,327/28, issued July 24, 1929, applied for August 28, 1928, in Japan, November 16, 1927, claims powdered insecticide prepared by causing the active ingredients of the root of derris species to be absorbed by an earth or earths such as clay, china-clay, earthenware clay or kieselguhr, which is or are free from water, alkali, and like substances detrimental to the active ingredients, adding thereto a suitable quantity of magnesium carbonate, calcium carbonate, or like adhesive medium, and crushing the resultant mixture to powder.

Examples are (1) twenty-five grams of powder of powdered dry roots of derris are extracted with a half pound of ether, and, while the liquid is agitated, 150 grams of dry kieselguhr are added. The ether is then recovered. This kieselguhr containing the absorbed active ingredient is mixed with 2 grams of powdered sulphur, 2 grams of powdered resin, and 45 grams of magnesium carbonate. The mixture is then finely powdered by a crusher. (2) Fifty grams of raw root of derris are crushed and pressed, and the juice is collected. This operation is repeated two or more times. To the juice, thus obtained, 150 grams of reddish earth is added, and the water content of the mixture is entirely driven off as by a vacuum evaporator under low temperature. This earth containing the absorbed active ingredients is mixed with quantities of powdered sulphur, powdered resin, and magnesium carbonate as in the previous example. The resultant mixture is then finely powdered by a crusher.

The Imperial Chemical Industries of Australia and New Zealand Ltd. (45), in Australian patent 20,894/35, issued February 14, 1935, applied for January 11, 1935, claims a method of treating sheep or other animals in order to render them immune from attack or infestation by blow flies or the like, which consists in subjecting the animals to treatment with a solution or a suspension containing one of the xanthates. An insecticidal agent such as a silicofluoride, derris, pyrethrum, etc., may be added. One of the claims reads: "A new article of manufacture for the purposes indicated, produced by mixing together the following substances substantially in the proportions indicated:--

potassium ethyl xanthate	8 parts
derris root powder	4 parts
bentonite	2 parts
sulphonated cod oil	1 part"

British Patents:

McDougall, McDougall, and McDougall (63), in British patent 3,204, issued May 8, 1912, applied for February 8, 1911, claim in the manufacture

Correction for E-446

The following information should be inserted on page 22 after the 3 lines at the top of the page. These patents, although in E-402 - (Tephrosia as an insecticide - a review of the literature, Feb. 1937) and two of which are also referred to on page 17 of the present publication were inadvertently omitted from the abstracts of United States Patents, beginning on page 21.

Lemmens and Fryer, in United States patent 1,242,954 issued October 16, 1917; applied for January 16, 1917; claim a parasitocidal preparation comprising comminuted parts of Tephrosia, and a soap vehicle.

"In the manufacture of a sheep dip, in accordance with the present invention, the seeds, leaves, and other portions of the plant are ground to powder and if desired mixed with other ingredients. In the manufacture of an insecticide for use for the destruction of plant pests, the ground material may be mixed with soap, sulphur, or other substances. The said ground material may also be mixed with various other substances to form preparations for the destruction of earthworms, grubs, and the like. The compound contained within the material is extractable therefrom, when in a finely divided state, by moisture or by immersing the material in water. The said compound when extracted then mixes with the substances which may be added to the ground material to produce the desired finished product ready for use for dipping or spraying." Softening reagents, antiseptics or bactericides may be added thereto.

Lemmens and Fryer, in United States patent 1,242,955 issued October 16, 1917; applied for January 16, 1917; claim a new anti-parasitic compound comprising a benzine extractive from desiccated parts of the plants known as Tephrosia, mixed with an emulsifying agent.

"In the manufacture of sheep dip such compound is mixed with soap, sulphur, or other ingredients and made into a paste, powder, or liquid in any known manner. Likewise, in the manufacture of an insecticide for use for the destruction of plant pests, the compound aforesaid may be mixed with soap, sulphur, or other substances which serve as emulsifying or carrying agents and diluents.**** The dip, wash, or the like may contain any desired proportion of the compound, according to the class of service to which it is to be put. Or it may be let down or diluted before use. In practice, the mixture employed will contain from 1-1/2 ounces of the compound to 100 gallons of water for the destruction of small and delicate insects up to 12 ounces of the compound to 100 gallons of water for the destruction of the more hardy and vigorous varieties."

(over)

Grove, in United States Patent 1,455,485 issued May 15, 1923; applied for July 26, 1922; claims a medical compound for the treatment of diabetes, including a liquid preparation of Tephrosia virginiana and a lithia salt in solution, T. virginiana being in such proportions as to cause it to reduce the sugar content and insipidity of the urine and the lithia salt to activate as a solvent and diaphoretic. The preferred formula is T. virginiana (ground roots), 9 ounces; lithii citras, 1 ounce; powdered cinnamon, 30 grains; cochineal, 3 grains; alcohol, 2 ounces; and water, 16 ounces. In preparing this remedy the roots of the herb T. virginiana, dried and powdered, are boiled with 1 pint of water for about 1 hour. The decoction is then cooled and strained. The other ingredients are then added, and the compound is allowed to stand for 2 days. It is then filtered and bottled. The compound is to be taken internally, and under ordinary circumstances a dose is 2 teaspoonsful every 3 hours. [From his references to the literature relating to the medicinal properties of this plant, which he calls goatsrue and also Tephrosia virginiana, it is probable that Grove confused T. virginiana with Galega officinalis, as both are commonly known as goatsrue. R. C. R.]

of insecticides, sheep dips, animal washes and vermifuges, and the like, the use of an organic salt or salts, compound or compounds obtained from the plants, bushes, or trees known as tuba rabut, tuba todong, nero (or akar) tuba, and perkakal, as set forth. The powdered bark, root, or other portions of the plant are extracted by a suitable solvent, such as benzine, the excess of solvent is evaporated, and the gummy residue is then mixed with the necessary ingredients for producing the required dip, wash, insecticide, or vermifuge, or it may be crystallized before such use. During the manufacture of the dip, a suitable water-softening reagent or a suitable antiseptic, bactericide, or coloring matter may be added. In practice the mixture employed will contain from 1 ounce of the compound to 100 imperial gallons of water for the destruction of small and delicate insects, up to 12 ounces of the compound to 100 imperial gallons of water for the destruction of the more hardy and vigorous caterpillars. A suitable apparatus for the extraction is described. The extract may be mixed with sulphur, soap, or other ingredients and made into a paste, powder, or liquid in any known manner.

McDougall, McDougall, and McDougall (65), in British patent 10,215 of 1911, issued May 28, 1912, applied for April 27, 1911, claim in the manufacture of sheep dips, animal washes, vermifuges, and insecticides, the use of an organic salt or salts, compound, or compounds obtained from the plants, bushes, or trees of the family known botanically as Derris of the order of Leguminosae, as set forth. This invention is an improvement over that described in British patent 3,204 of 1911. The mixture may be used for destroying earthworms and as a dose for animals for the destruction of internal parasites. "As a dose the mixture may contain from about 1/2 to 1 ounce of the extract emulsified with about 4 ounces of soapy matter, the whole being then made up to 20 ounces by the addition of sulphur."

McDougall, McDougall, and McDougall (64), in British patent 8,322 of 1912, issued December 19, 1912, applied for April 6, 1912, claim improved insecticides, sheep dip, vermifuges, and the like comprising, in a powdered or finely divided form, the root, bark, or other portions of the plants, bushes, or trees of the Dalbergiace tribe and Derris family and of perkakal, from which an organic salt or salts, compound, or compounds is extractable by moisture or water, substantially as described. This invention is an improvement over that described in the patentees' prior specifications--British patents 3,204 of 1911 and 10,215 of 1911. The ground derris root may be mixed with soap, sulphur, or other substances.

Lommens and Fryer (54), in Britain patent 105,525, issued April 19, 1917, applied for December 4, 1916, claim

"In the manufacture of insecticides, sheep dips, animal washes and the like, the use of an organic salt or salts, compound or compounds obtained from the seeds, leaves and other portions of the plant of the species and varieties of the genus of plants known as *Tephrosia* of the natural order Leguminosae and the sub-order Papilionaceae, particularly the species of *Tephrosia* known as *Tephrosia vogelii*."

An apparatus for extracting the powdered dry plant material with benzine is described. In practice, the mixture employed will contain from 1-1/2 ounces of the compound to 100 imperial gallons of water for the destruction of small and delicate insects up to 12 ounces per 100 gallons of water for the destruction of the more hardy and vigorous varieties.

Bagnall-Bull (4), in British patent 214,822, issued May 1, 1924, applied for April 16, 1923, claims a method of obtaining derris extract for use as an insecticide, vermifuge, sheep dip, or the like, which consists in breaking or crushing the fresh roots or other portions of the plants of the family known botanically as Derris or Tuba, treating them with water so as to obtain a milky emulsion or suspension of the juices containing toxic properties, and then separating the emulsified substance from the whole or a part of the water.

To separate the emulsified substance from the water, evaporate the whole or a part, preferably in vacuo, thereby obtaining a paste of a consistency suitable for transport, or add to the milky emulsion milk of lime, which coagulates or precipitates the emulsified substance. Free the precipitate obtained by the last method from excess moisture by evaporation, by filtration, or by centrifugalizing. Or mix it with tartaric acid or any other suitable acid in quantity equivalent to the amount of lime contained in the precipitate. Or the milky emulsion may be rendered alkaline with soda carbonate, to which is then added a brine or salt solution in a quantity sufficient to salt out the emulsified substance, which then precipitates. The emulsified substance or precipitate so obtained is then dried and ground or can be used in a moist condition according to the use to which it is to be applied. Grinding produces a powder, which by reason of its fineness admits of a suspension of the drug in water. This preparation alone or mixed with inert powder may be used as a dusting powder, or it may be mixed with soap, sulphur, or other suitable substance for use as insecticidal washes, sheep dips, or for spraying, either as a powder or when mixed with water.

McDougall and Howles (58), in British patent 226,250, issued December 23, 1924, applied for June 23, 1923, claim a process for the production of an insecticide, sheep dip, vermifuge, or the like which is directly miscible with water, by crushing or pressing the root portion of plants, bushes, or trees of the Dalbergia tribe and Derris family to a pulp, scouring the fibrous particles by mechanical means in the presence of water and finally separating the fibrous material from the magma, substantially as described. The latex may be evaporated in a vacuum at a low temperature. The active material obtained as before described, may, if desired, be mixed with water and/or soaps of various kinds, such as resin, fatty acid or sulphonated oil soaps, sulphur, antiseptics, water softening materials, spreaders, and the like, for the destruction of insect pests on animals and/or plants or for internal or external parasites. The active ingredients may be separated from the latex by precipitation with a basic salt of aluminum, iron, or lead. For example, to 100 cubic centimeters of the latex, a quantity of concentrated aluminum sulphate solution is added, so that the final dilution of the aluminum sulphate in the latex is equivalent to 0.2 percent. To this is added a solution of caustic soda in quantity insufficient to precipitate the whole

of the aluminum sulphate as hydronide. The coagulum is then treated with an appropriate dilute acid to free it from metallic salts, after which it is readily emulsified in water and soap.

McDougall and Yalding, Ltd. and P. J. Fryer (69), in British patent 229,773, issued March 2, 1925, applied for December 1, 1923, claim insecticides, sheep dips, animal washes and the like, comprising an organic salt or salts, compound or compounds obtained from the root, bark or other portions of the plants, bushes, or trees of the Dalbergiace tribe and Derris family and/or of perkakal, and sulphonated castor oil as a vehicle or spreading or activating agent for such organic salt or salts, compound or compounds.

McDougall and Howles (60), in British patent 233,857, issued May 21, 1925, applied for April 15, 1924, claim in insecticides, the combination comprising a mixture of nicotine with the aforesaid derris extract or powdered derris plant and a neutral inert body, substantially as described. An example of preparing this insecticide is: 2 parts by weight of nicotine are mixed with 5 parts by weight of powdered root and 93 parts of precipitated calcium carbonate. The extract may be obtained by the process described in prior specification Nos. 3,204 of 1911, 10,215 of 1911, and 8,322 of 1912, or in specification No. 226,250. This is used in powder form for dusting hops and fruit trees for the destruction of aphids, psylla, and various caterpillars.

The Zaidan Hojin Rikagaku Kenkyujo (102), in British patent 239,483, issued May 6, 1926, applied for April 24, 1925, in Japan, September 8, 1924, claims a process of preparing a liquid insecticide containing the effective ingredient of the derris species, comprising the step of preparing a primary reagent in which the effective ingredient is mixed with one or more oils and/or fats, waxes, resins, higher alcohols, higher hydrocarbons, higher fatty acids (including Turkey red oil), which are entirely free from water and alkali; and the step of preparing a secondary reagent in which the primary reagent is mixed as a colloidal solution, substantially as described. Examples are:

"(1) A solution of 10 litres of ether containing 10 grammes of rotenone is added to a mixture of 400 grammes of fish oil with 40 grammes of hydrogenated oil. After sufficient agitation, the greater part of the ether is recovered by evaporation and condensation, and thereby a semi-solid mixture is obtained as a primary reagent. Five hundred grammes of soap are mixed with 100 kilogrammes of water to prepare a colloidal aqueous solution as a secondary reagent. In use, the primary reagent is mixed with the secondary reagent and it may be suitably diluted with addition of 100 kilogrammes of water when used as an insecticide. The secondary reagent may be prepared at any time by users.

"(2) Ten liters of ether are added to 1 kilogram of the derris root. After 4 days, the extract is added to 400 grammes of chrysalis oil and 100 grammes of lard. After evaporating the ether a semi-solid substance is obtained as a primary reagent. The pre-

paration of the secondary reagent may be effected as for Example - 1.

"(3) Twenty liters of petroleum ether are added to 1 kilogramme of derris root. After 4 days, the extract is added to 200 grammes of beeswax and 50 grammes of pine resin. The larger portion of the petroleum ether is recovered by evaporation and condensation and thereby a semi-colloidal substance is obtained as the primary reagent. The secondary reagent may be prepared by adding a colloidal solution in which 200 grammes of soap are dissolved in 100 kilogrammes of water."

"It is to be understood that substances containing rotenone is intended to mean any pure or impure substance obtained from raw root of the derris species, solutions of the substances in one or more solvents such as methyl alcohol, absolute alcohol, ether, petroleum ether, chloroform, tetrachloride of carbon, and extracts of root of derris with such solvents. The solvents are recovered during the process. By oils it is intended to mean all kinds of animal oils such as sardine oil, herring oil, cod liver oil, shark oil, chrysalis oil and all kinds of vegetable oils such as cocoa-nut oil, bean oil, rapeseed oil, castor oil and the like excluding Turkey red oil. By fats it is intended to mean all kinds of animal fats such as lard, beef tallow, hydrogenised animal oil and hydrogenised vegetable oil. By waxes it is intended to mean wood wax, beeswax and the like, while resins include pine resin, copal and the like. By higher hydrocarbons paraffin and the like is intended, while higher alcohol includes glycerine and the like. These substances are selected as desired. Colloidal aqueous solution includes a solution of soap, glue, egg-albumin, bean milk, dextrine, starch, bengal isinglass, animal albumin and the like. There may be added to said aqueous solutions a small quantity of methyl alcohol, ethyl alcohol, chloroform, ether, camphor oil, lemon oil, and other solvents. Said additional substances may be added after they are dehydrated to the oils or fats used in preparing the rotenon-containing reagent. Ordinary water can be also employed in place of a colloidal aqueous solution."

McDougall and Yalding Ltd. and P. J. Fryer (70), in British patent 246,252, issued January 28, 1926, applied for November 12, 1924, claim an improvement in or modification of the invention claimed in specification no. 229,773, according to which the sulphonated castor oil is replaced by any other sulphonated fatty oil, sulphonated sperm oil or a sulphonated wax. The most satisfactory results are obtained by extracting derris with benzene or other suitable volatile solvent, and after the solvent has been distilled off, by dissolving the residue by the aid of gentle heat in the sulphonated oil or wax. The best proportion is 1 part of derris extract to 10 to 50 parts of the sulphonated oil or wax. This mixture may be used with lead arsenate and Bordeaux mixture, but for use with lime-sulphur solution it is necessary to add from 5 to 20 percent of casein to prevent precipitation. Powdered casein may be dissolved in the sulphonated oil by gently warming and stirring.

The Japanese firm Nittai Sangyo Kabushiki Kaisha (76), in British patent 247,140, issued August 5, 1926, applied for June 25, 1925, in Japan February 4, 1925, claims a process for obtaining insecticidal or other toxic substances from plants of the Derris species in solid form, consisting in collecting fresh juice of crushed fresh roots of the plants with the addition of water, evaporating the juice at a temperature not exceeding 70° C. and drying the same into a solid state, removing surplus resinous matters from the juice, substantially as described.

The fresh root of tuba is crushed, and after a small quantity of water is added it is filtered. The filtrate is allowed to settle for about three hours in order to allow resinous matters to separate. The supernatant material is then evaporated in a vacuum evaporating pan or an ordinary evaporator at a temperature not exceeding 70°. The contents when pasty, are spread on glass or iron plates and dried in a drying chamber at a temperature of 40° to 45° until they become a deep-brown, solid mass. The solid mass may be pulverized for convenience of use. The powdered product is a light-brown powder which contains about 30 to 32 percent of rotenone, 34 to 35 percent of derrid, and 35 percent of impurities, such as resin, albumen, gum, and ash. "When it is properly diluted by the addition of the desired quantity of water, it becomes the best insecticide for agricultural purposes."

"The product is more effective than those containing the same quantity of a single kind of toxin, because, as is well-known, the simultaneous use of two or more kinds of toxins is far more effective than the use of the same quantity of any one of them."

McDougall and Yalding Ltd. and P. J. Fryer (71), in British patent 280,256, issued November 17, 1927, applied for November 24, 1926, claim a process for producing concentrated complete and self-contained insecticides containing physiologically active, substantially water-insoluble plant extracts, extracted from plants mainly of the order Leguminosae (particularly those containing tuba-toxin (rotenon) and or tephresin) which consists in dissolving such extracts in a phenolic body or suitable solvent, adding the solution to a hydrocarbon oil or mixture of hydrocarbon oils and finally emulsifying the whole, the emulsion so produced necessitating only simple admixture with water in suitable proportions to form a spraying solution.

According to one example, 1 part of the resinous active principle from derris or other source is dissolved by warming it with 2-1/2 parts of commercial cresylic acid. This is now added to 288 parts of a suitable hydrocarbon oil--for example, medium lubricating--oil fraction of American petroleum--and after the addition of 100 parts of a 10 percent glue solution the whole is emulsified. According to a second example, the solution of the active principle in cresylic acid, as described in the first example, is added to 288 parts of suitable hydrocarbon oil, and the whole is mixed with 12 parts of ammonium oleate or linoleate to form a homogeneous solution, which is diluted with water.

The Zaidan Hojin Rikagaku Kenkyujo (108), in British patent 285,797 issued November 29, 1928, applied for February 20, 1928, in Japan February 18, 1927, claims a process for producing a liquid insecticide containing the effective ingredient of derris species, which comprises forming a primary material in which the effective ingredient is mixed with an anhydrous and alkali free vehicle for the active ingredient, said vehicle consisting of one or more oils, fats, waxes, resins, higher alcohols, higher hydrocarbons, higher fatty acids, together with a substantially equal quantity of the anhydrous solvent, benzol, carbon tetrachloride or trichlorethane, which primary material is subsequently to be mixed with an aqueous soap solution to form a colloidal solution.

Examples are: (I) To 100 grams of powdered dorris root, 10 liters of ether is added and thereby the effective ingredient of the dorris root is extracted, the whole or greater part of the ether is evaporated and recovered. To the residual effective ingredient a mixture of 150 grams of anhydrous and alkali-free fish oil (as a vehicle) and 150 grams of benzol (as the anhydrous solvent) is added, so that a solution of the effective ingredient is thus prepared. This is the improved primary material. The soap solution is easily prepared by, for instance, adding 500 grams of soap to 100 kilograms of water. The solution thus obtained is mixed with the improved primary material in use.

(II) To 1 kilogram of powdered dorris root, 5 kilograms of benzol is added and the effective ingredient of the dorris root is extracted while warming the benzol. The benzol is recovered by evaporation. To the residual effective ingredient, a mixture of 600 grams of anhydrous and alkali-free fish oil (as a vehicle) and 600 grams of carbon tetrachloride (as an anhydrous solvent) is added so that a solution of the effective ingredient is formed. This is the improved primary material. The soap solution employed therewith may be the same as in the above mentioned example. Trichlor-ethane $C_2H_3Cl_3$ as the anhydrous solvent can also be used. Also in the two examples given, the improved primary material can be easily prepared in the form of powder when 900 grams of anhydrous gum arabic are added.

The Zaidan Hojin Rikagaku Kenkyujo (109), in British patent 300,606, applied for July 17, 1928, in Japan, November 16, 1927, made essentially the same claims as in Australian patent 15327/28 (110).

The Standard Oil Development Company (95), in British patent 350,897, issued June 15, 1931, applied for February 15, 1930, claims an insecticidal, insect-repellent, or fungicidal composition comprising petroleum white oil, an extract of a plant having insecticidal properties and an emulsifying agent. The insecticidal plant may be a fish poison such as Dorris, Lonchocarpus, or Tephrosia. Instead of, or together with the fish poison pyrethrum may be used. An example is "Two kilograms of ground dorris roots were extracted with four litres of benzol. Distilling off the solvent in vacuo 113 grams of dry extract were obtained representing 5.65 percent of the dry dorris roots and having the appearance of a reddish brown resin. Eighty grams of this extract were fed into a colloid mill with a solution of 750 grams of oil-soluble sodium sulphonate derived from petroleum oil in 2.5 litres of technically pure white oil. Water was also added in the amount of 1.5 litres. A thick emulsion resulted similar in appearance to mayonnaise and ready for marketing. Part of this preparation was diluted with 50 times its weight of water and gave an effective insecticidal spray for horticultural purposes." Dorris may be extracted with a volatile solvent such as benzol or isopropyl alcohol or with white oil directly. Volatile solvents such as alcohols, ethers, esters, ketones, aromatic hydrocarbons, etc., may be used to increase its solvent power. It is desirable to distil off subsequently most of the volatile solvent from the product obtained. Another class of solvents may also be added to the white oil before extraction, namely, steam distilled pine oil, terpineol, phenol,

eresol, cyclohexanol and its homologues, and hydrogenated or chlorinated naphthalenes such as the material known under the trade name "halowax oil". The ingredients just mentioned are not removed by subsequent distillation. They are desirable in the product and confer upon it a fungicidal and bactericidal effect in addition to its insecticidal properties; they also facilitate the emulsification.

Various emulsifying agents may be used such as, for instance, fatty acid soap, rosin soap, saponin, soaps of petroleum sulphonic acids, soaps of sulphonated vegetable or animal oils, etc. When the plant is extracted directly with the white oil, part of the soap used may be added to the white oil before the extraction since it has been found that the presence of a soap facilitates the extraction. In many cases it is preferred to use the soap of an oil-soluble sulphonic acid derived from petroleum. Such acids are obtained in the refining of the white oils. This spray is effective against various aphids, tent caterpillars, scale insects, red mite, tomato moth, cabbage worm, sawflies, etc. The oxidizing action of the air on white oils and the development of acidity may be inhibited by the addition to the oil of small percentages of some substance known generally under the name of anti-oxidation catalyst. As examples, phenanthrene and anthracene may be mentioned, 0.5 percent of which when added to the white oil preparation are sufficient to protect completely the latter against the oxidizing action of air. A great variety of other anti-oxidation catalysts may be used such as, for instance, diphenyl, toluidine, which have an oxidation inhibiting action when present in concentration as low as 0.3 percent of the oil. Other anti-oxidation catalysts which may be used are α -naphthol, camphor, nitroeresol, phenylhydrazine, furon, etc. The anti-oxidation catalysts are preferably added to the white oil containing vegetable insecticidal principle before the emulsification of the latter is carried out.

Graafland and Nellensteyn (30), in British patent 362,526, issued December 10, 1931, applied for September 10, 1930, in Holland September 12, 1929; claim an antifouling composition against growth of and damage by organisms in water comprising a hydraulic binder and a poisonous substance other than one which acts upon iron to cause formation of salts of iron, the composition being adapted to permanently retain its hydrophile character. A suitable composition is Portland cement, 100 parts by weight; powder of blossoms of Chrysanthemum cinerariaefolium, 5 - 30 parts by weight; water, 110 parts by weight. The Portland cement must be carefully mixed with the powder of said blossoms. The mixture is rendered plastic by the addition of a quantity of water, which plastic mixture is applicable in an easy manner, such as for instance by painting with a brush, by spraying, or the like. After hardening of the coating a porous layer has been formed, in which, because of its hydrophile character, the poison remains effective and prevents the organisms from settling on or in it. Another effective poison is for instance extract from Derris elliptica.

The I. G. Farbenindustrie, A. G. (39), in British patent 377,069, issued July 21, 1932, applied for November 27, 1931, in Germany November 27, 1930, claims a preparation for killing or eliminating flies comprising

an insecticidal substance and a product obtained by subjecting petroleum or a petroleum distillate to partial or complete hydrogenation so that the odor of the non-hydrogenated material is removed. For example, 0.4 part by weight of derris extract (obtained by extracting the roots of Derris elliptica Benth.) and 10 parts of diphenyl ether are dissolved in perhydrogenated petroleum boiling between about 180° and 290° C. and the solution is utilized in the usual manner for killing flies. When using extracts, as for instance extract of pyrethrum flowers, the said hydrogenated products may be used as solvents for preparing these extracts.

The I. G. Farbenindustrie, A. G. (41), in British patent 378,300, issued August 11, 1932, applied for December 12, 1931, in Germany December 13, 1930, claims a preparation for killing or eliminating flies comprising an insecticidal substance and a product obtained by partial or complete hydrogenation of a mineral oil or a distillation product thereof, except petroleum or a petroleum distillate, or a product of the same kind consisting of a synthetic oil or a distillation product thereof. Instead of using the hydrogenated petroleum or petroleum distillates mentioned in British patent 377,069, favorable effects are obtained by using the products obtainable by subjecting other oils to partial or complete hydrogenation, so that the odor of the non-hydrogenated material is removed. For the preparation of agents for killing flies there may, for instance, be used the hydrogenation products of mineral oils (for instance lignite-tar oil, coal-tar oil or shale oil) or the hydrogenation products of distillation products thereof, or there may be used synthetic oils of the same kind obtained, for instance, by the hydrogenation of brown coal until the hydrogenation products are free from unpleasant odor or there may be used the distillation products of the oils obtained by such hydrogenation. There may advantageously be used hydrogenated oils of a specific gravity of about 0.75 to 0.85 at 20° C. and boiling between about 100° C. and about 300° C.; such hydrogenated products may be obtained by hydrogenating, under raised pressure and temperature in the presence of a suitable hydrogenation catalyst, lignite-tar oil, coal-tar oil, shale oil, solvent naphtha and similar oils.

The insecticidal substance may be pyrethrum extract, derris extract or an ester or other of benzoic acid or a derivative or substitution product of this acid. For example 0.5 part of pyrethrum extract, 3.5 parts of 5-chloro-2-hydroxybenzoic acid amyl ester and 2 parts of iso-bornyl acetate are dissolved in hydrogenated solvent naphtha. The preparation completely kills flies, culicidae, and similar insects.

The Standard Oil Development Co., assignees of Sankowsky and Fulton (96), in British patent 394,977, issued July 4, 1933, applied for January 4, 1932, in the United States January 5, 1931, claim an improved insecticide comprising hydrocarbons rich in "unsaturated hydrocarbons" and obtained by the extraction of a petroleum naphtha with a solvent having a preferential solvent action for said "unsaturated hydrocarbons" in admixture with an insecticidal plant extract. The insecticidal plant

extract may be from pyrethrum, cube, derris, or the like. Tests upon houseflies showed that unsaturated hydrocarbons are more toxic than saturated ones.

The Chemische Fabrik Marionfelde G.m.b.H. (14), in English patent 432,626, issued July 30, 1935, applied for January 11, 1935, in Germany, January 18, 1934, claim "a process for the manufacture of durable, highly concentrated derris root extracts which are emulsifiable with water, characterized by adding weakly acid alkali resin soaps to a derris root extract obtained by means of, and containing, di- or trichloroethylene."

An example of the process is as follows:

"Derris roots are exhaustively extracted with di- or trichloroethylene, and the filtered solution is concentrated until it contains one part of derris root extract in one part of di- or trichloroethylene. Resin is saponified with caustic soda solution of 36° Be, only sufficient caustic soda solution being added for the soap formed to have a pH value of 5.0 or less. In this way, there is formed a weakly acid resin soap having a water content of only about 20%. Fifty kilograms of this soap are dissolved in 50 kilograms of dichloroethylene, while being heated under a reflux condenser, and the solution is mixed with 100 kg. of the derris root extract concentrated to 1:1."

Pyrethrum extract may be similarly used. Di- and trichloroethylene are better solvents for derris constituents than are alcohol, benzene, carbontetrachloride, etc.

Wikstrom (102), in British patent 434,660, issued September 6, 1935, applied for March 19, 1934, claims an insecticide comprising cymene [cymol] or a derivative thereof and the active constituents of pyrethrum or derris or quassia or equivalent vermin-destroying agent of vegetable origin or a combination of any two or more of said agents, the cymol or its derivative serving as a solvent or carrier for the said agent or agents. The derris may be extracted with cymene.

A further very valuable ingredient is chlorinated or sulphurated etheral oils. These oils act as contact (external) poison and as internal poison and further have very powerful paralyzing effects, thereby facilitating the action of the poison or poisons present. These products may be prepared by treating an etheral oil, such as oleum citronellae, oleum eucalypti (for example the product known as "Globulus"), oleum cedri, preferably in heat and with stirring with aqua regia, chlorine water, liquid chlorine, sulphuric acid, sulphur chloride, sulphides or polysulphides such as the alkali sulphides. The product obtained is washed with water, which may be alkaline, and is then dissolved in cymene (or some other suitable solvent).

A further valuable addition is a soap, preferably a resinous soap and preferably a soap which is not completely saponified, so as to retain a certain degree of stickiness and prevent the vermin from moving while

the poison is acting. It further serves as an attenuating agent and as an emulsifying agent.

It may also be of advantage to add a specific emulsifying agent, for example, triethanolamine.

Examples are: (1). Derris-cymol extract (15 percent) 1 part, Triethanolamine 0.1 part, water 100 parts. The triethanolamine is first dissolved in water and the derris-cymol extract is then added with stirring. The rest of the water is then added. The preparation is facilitated if heat is used. (2). Pyrethrum-cymol extract (15 percent) 1 part, derris-cymol extract 1 part, water 200 parts. (3). Pyrethrum-cymol extract (15 percent) 1 part, derris-cymol extract 1 part, quassia-cymol extract 1 part, water 300 parts. (4) Pyrethrum-cymol extract 15 percent 1-60 parts, derris-cymol extract or quassia-cymol extract 1-60 parts, chlorinated or sulphurated ethereal oils 2-60 parts, soap 200-400 parts, water 2,000-200,000 parts.

A convenient stock solution may consist of: Pyrethrum-cymol extract (15 percent), derris-cymol extract or quassia-cymol extract total 15 parts, chlorinated or sulphurated ethereal oils, 6 parts, soap 79 parts. To this mixture is added, when it is to be used, enough water to make a 0.25-15 percent solution. The liquid may be sprayed or sprinkled on the plants or other objects which are to be treated, but dipping or other methods of distributing the liquid may also be employed.

The Booth Steamship Co. Ltd. and H. G. Ward, (7), in British patent 437,171, issued October 24, 1935, applied for March 30, 1935, claim:

The process for extracting toxins for the manufacture of insecticides from roots containing rotenone, according to which the roots in crushed or ground form are digested in common alcohol acidulated by pure sulphuric acid, and the mixture filtered so that the final filtrate contains the rotenone and incidentally also other toxins of an insecticidal value present in the root.

The filtrate may be neutralized with sodium carbonate and refiltered. The final filtrate may be mixed with soft soap so as to obtain a semi-solid product which can be diluted with water before use.

Fawcett and Imperial Chemical Industries, Ltd. (26), in British patent 446,576, issued April 29, 1936, applied for October 29, 1934; claim a process for the production of rotenone and/or preparations having insecticidal value and containing rotenone, which comprises subjecting the natural substances containing rotenone, to distillation at raised temperature in a high vacuum and collecting the distillate. A vacuum of between 10^{-2} and 10^{-6} mm. of mercury is used. The condensate from the distillation may suitably be removed from the condensing surface by melting down, by scraping, or by rinsing with a solvent. An example of the process

is as follows: Fifty parts of dry ground derris root are mixed with 100 parts of a linseed oil stand oil which has previously been treated in a high vacuum at 220° C. to remove volatile constituents. The mixture of derris root and oil is fed on to a heated surface or surfaces maintained at about 120° C. and in close proximity to a cold condensing surface, the whole apparatus being evacuated to about 10⁻⁴ mms. of mercury. A pale yellow condensate is obtained, giving the reactions of rotenone and containing substantially all the active material present in the original root.

Other carrier liquids such as Apiezon, heavy mineral oil, cottonseed oil, olive oil, etc., may be used. "When it is desired to obtain a solution of the active ingredients as distillate, the dry powdered derris or cube root may be mixed with a carrier liquid which is either wholly distillable at the temperatures and pressures employed, or comprises a portion so distillable. Thus a mixture of the oils known respectively under the trade names 'Apiezon A' and 'Apiezon K' may be used. The active ingredients distil over together with the 'Apiezon A' leaving behind a suspension or solution of the residue in the 'Apiezon K'. The distillate, which is a more or less concentrated solution of the rotenone etc. in 'Apiezon A' (the concentration can be varied by varying the proportion of 'Apiezon A' taken), is suitable for dilution with kerosene or other volatile mineral oil for use as insecticidal spray."

Canadian patents.

McDougall and Howles (61), in Canadian patent 247,627, issued March 17, 1925, applied for May 22, 1924, make the same claims as in British patent 226,250.

McDougall and Yalding Ltd., assignee of P. J. Fryer (67), in Canadian patent 253,319, issued September 1, 1925, applied for November 26, 1924, claim a process for the production of insecticides, sheep dips, animal washes, and the like which consists in mixing or incorporating with an organic salt or salts, compound or compounds obtained from the root, bark, or other portions of the plants, bushes, or trees of the Dalbergiace tribe and Derris family and/or of perkakal, a sulphonated fatty oil, sulphonated sperm oil, or a sulphonated wax as a vehicle or spreading or activating agent for such organic salt or salts, compound or compounds. The sulphonated fatty oil may be sulphonated castor oil.

Dennis (19), in Canadian patent 223,233, issued September 24, 1929, applied for March 12, 1929, makes essentially the same claims for ground cube root with the fibrous element removed and for an extract of cube root as he previously made it in his United States patents.

The Standard Oil Development Company, assignee of Grant (31), in Canadian Patent 333,642, issued June 27, 1933, applied for October 25, 1929, makes the following claims:

"A spray for sensitive foliage comprising a derris extract incorporated in white oil. A composition of matter comprising an aqueous emulsion of white oil containing an organic solvent extract of derris. A spray comprising a concentrated aqueous emulsion of white oil containing rotenone diluted with about 50 to 100 times its volume of water."

The Standard Oil Company, assignee of Adams (1), in Canadian patent 338,896, issued January 23, 1934, applied for October 23, 1931, claims a mothproofing composition comprising a chlorinated hydrocarbon extract of cube. It also claims a mothproofing composition which comprises a chlorinated hydrocarbon extract of cube dissolved in petroleum naphtha having a boiling range of about 450-550° F.

The Standard Oil Company, assignee of W. J. McGill (73), in Canadian patent 338,897, issued January 23, 1934, applied for April 1, 1932, claims a mothproofing solution containing an extract of derris. Claim is also made for a solution of the active principle of haiari in petroleum naphtha boiling between 450-550° F.

The Standard Oil Development Co., assignee of Sankowsky and Fulton (87), in Canadian patent 340,315, issued March 20, 1934, applied for December 24, 1931, in the United States January 5, 1931, claim an improved insecticide, comprising hydrocarbons rich in unsaturated hydrocarbons and obtained by the extraction of a petroleum naphtha with a solvent having a preferential solvent action for said unsaturated hydrocarbons, and containing the extractives of at least one of the plants selected from the group consisting of pyrethrum, cube, derris, and the like.

The International Hydrogenation Patents Company, Ltd., Vaduz, Liechtenstein, assignee of Pfaff and Dunkel (81), in Canadian patent 345,383, issued October 16, 1934, applied for December 12, 1931, in Germany December 13, 1930, claim an insecticidal composition, comprising a solution of an insecticidal plant extract and/or a compound of the group comprising esters and others of benzoic acid and substitution products thereof in a hydrogenated mineral oil boiling between about 100° C., and 300° C. and having a specific gravity of about 0.75-0.85 at 20° C.

Schotte and Görnitz (88), in Canadian patent 348,379, issued February 26, 1935, applied for January 24, 1934, claim an insecticidal preparation consisting of a mixture containing extracts of drugs containing rotenon and veratrin. They also claim an insecticidal preparation consisting of a mixture containing radix derridis ellipticae and semen sabadilla.

Schotte and Görnitz (89), in Canadian patent 348,380, issued February 26, 1935, applied for January 24, 1934, claim a solidified finely distributed mixture of a molten carrier and finely distributed derris root. Powdered tobacco or pyrethrum may be used in place of the derris.

The Kessler Chemical Corp., assignee of S. Goldschmidt and K. Martin (29), in Canadian patent 357,959, issued May 19, 1936, applied for December 17, 1934, in Germany December 27, 1933, claim as a toxic agent and more particularly an insecticide, a preparation comprising as an active constituent an aromatic isothiocyanate in which the isothiocyanate radical is directly attached to the aromatic nucleus, in mixture with another insecticide such as nicotine, derris extract, or rotenone, respectively.

Ward (101), in Canadian patent 362,903, issued December 22, 1936, applied for August 29, 1935, in England, March 30, 1935, makes essentially the same claims as in British patent 437,171 issued October 24, 1935, and in United States patent 2,056,438 issued October 6, 1936.

The Shell Development Company (92), assignee of W. J. Yates, in Canadian patent 369,390 issued October 19, 1937, applied for December 24, 1936, in the United States January 6, 1936, claims an insecticidal composition comprising a stable mineral oil distillate, an oil-soluble vegetable insecticide selected from the class consisting of rotenone and active ingredients of pyrethrum, and a small proportion of a substituted aryl compound in which at least one hydrogen atom of the aromatic nucleus is replaced by a member of the group consisting of amino, hydroxyl, and alkyl radicals. Dihydroxy and dialkyl mono-hydroxy aromatic compounds are also included among the substituted aryl compounds.

Imperial Chemical Industries Otd. (46), assignee of Fawcett, in Canadian patent 369,499, issued Oct. 26, 1937, applied for Oct. 28, 1935, claims the process for the production of rotenone preparations which comprises subjecting a natural rotenone-containing substance to distillation without ebullition at a pressure of 10^{-2} to 10^{-6} mms. of mercury under such conditions that the evaporating and condensing surfaces are substantially co-extensive and are in close proximity to one another, and collecting the distillate. The material may be dried and ground and mixed with a carrier liquid which is substantially volatile at the temperature and pressure to be employed. This is similar to British patent 446,576; French patent 797,052 and United States patent 2,096,678.

Dutch patents.

The Japanese firm Nettai Sangyo Kabushiki Kaisha (77), in Dutch patent 21,841, issued May 15, 1930, applied for February 10, 1926, claims an insecticide whose activity is due to the active principle of derris root, obtained by extraction with an organic solvent, and made by mixing the extract with one or more kinds of oils, fats, waxes, resins, higher alcohols, higher hydrocarbons, higher fatty acids (not including sulphonated oils or fatty acids) which are absolutely free from water or alkali. Examples are: (1) A solution of 10 grams of rotenone in 10 liters of ether is mixed with about 400 grams of fish oil and 40 grams of hard oil. After complete mixing a large part of the ether is recovered by evaporation and condensation. The volume is reduced to about one-half. Before using, 500 grams of soap are dissolved in 100 kilograms of water and into this is mixed the reduced portion above. Finally, the mixture can

be diluted with 100 kilograms water. (2) To 1 kilogram of derris root is added 10 liters of ether. After four days 400 grams of chrysalis oil and 100 grams of hog suet are added to the extract. The ether is evaporated leaving about one-half the original volume. From here on proceed as in Example No. 1. (3) One kilogram of derris root is soaked with 20 liters of petroleum ether. After 4 days 200 grams of beeswax and 50 grams of pine resin are added to the extract. After this has dissolved as much as possible, the greater part of the petroleum ether is recovered by evaporation and condensation, leaving a semicolloidal mass. This residue can be dispensed as a colloid in a solution of 200 grams of soap in 100 kilograms of water.

Rotenone decomposes on long contact with water or with alcohol containing water. Further, the active principle is unstable in the presence of alkalis.

Suitable solvents for the extraction of derris are methyl alcohol, ethyl alcohol, ether, petroleum ether, chloroform, carbon tetrachloride, etc. As oils the following may be used: all sorts of animal oils, such as sardine oil, herring oil, cod liver oil, shark oil, chrysalis oil, as well as all kinds of vegetable oil such as oil of coconut, bean (soya), rape seed or linseed oil, etc. As regards fats, all sorts of animal fats may be used such as (hog) suet, beef tallow, as well as hardened plant oils. As waxes, may be used the following: Beeswax, wood wax, etc. As resins, pine resins, copal, etc. Suitable higher hydrocarbons may be paraffin or similar compounds, higher alcohols, such as glycerine, etc., the ones chosen depending on circumstances.

As emulsifying agents the following may be used; aqueous solutions of soap, glue, egg white, milk of soya bean, dextrin, starch, Bengal fish glue, animal protein, etc. Sometimes to the aqueous solutions are added a small quantity ethyl alcohol, chloroform, ether, oil of camphor, citron oil, or other solvents.

French Patents:

McDougall and Yalding Ltd. (68), in French patent 589,506, issued May 30, 1925, applied for November 22, 1924, in England December 1, 1923, make essentially the same claims as in British patents 229,773 and 246,252. A sulphonated wax or a sulphonated fatty oil (castor or sperm) may be used as the spreading agent with derris extract.

McDougall and Howles (62), in French patent 589,969, issued June 9, 1925, applied for May 14, 1924, in England June 23 and November 22, 1923, make the same claims as in British patent 226,250.

Dupre and Company (23), in French patent 769,445 published August 25, 1934; applied for February 28, 1934, claim an insecticide consisting of quassia extract, vegetable oil and rotenone (or extract of derris or of cube), plus sassafras oil. This is made by mixing 10 kilograms of quassia

extract (25%) plus about 4 kilograms of triethanolamine with 50 kilograms of vegetable oil (e.g. huile de arachide) plus 11 kg. oleic acid plus 1 kg. rotonone plus 100 grams sassafras oil. For use the mixture is diluted with from 100 to 150 times its weight of water.

The J. D. Riedel-E. De Haen A.-G. (82), in French patent 778,841 issued March 25, 1935, applied for September 24, 1934, in Germany September 30 and November 8, 1933, claim a process in which extracts of plant insecticides (e. g. extracts of Chrysanthemum cinerariaefolium and of varieties of Derris) are atomized by air under pressure to destroy insects. Aqueous emulsions of these extracts may be used.

The Booth Steamship Co., Ltd., and H. G. Ward (8), in French patent 794,206, issued February 11, 1936, applied for August 27, 1935, in England, March 30, 1935, make the same claims as in British patent 437,171.

The Imperial Chemical Industries Ltd. (44), in French patent 797,052, issued April 20, 1936, applied for October 29, 1935, in England October 29, 1934, make the same claims as in British patent 446,576.

German Patents:

Lemmens and Fryer (55), in German patent 321,317, issued May 29, 1920, applied for December 21, 1916, make essentially the same claims as in United States patent 1,242,955.

Lemmens and Fryer (56), in German patent 327,583, issued October 11, 1920, applied for December 22, 1916, make essentially the same claims as in United States patent 1,242,954.

The I. G. Farbenindustrie, A. G. (40), in German patent 555,117, issued July 18, 1932, applied for November 28, 1930, claims a fly spray made by dissolving a fly destroying material such as amyl salicylate, phenyl salicylate, pyrethrum extract, or derris extract in a partially or wholly hydrogenated petroleum or petroleum distillation products. For example, 0.4 part derris extract (from roots of Derris elliptica) and 10 parts di-phenyl ether are dissolved in perhydrogenated petroleum.

Steindorff, Pfaff, Krämer, and Kern (97), assignors to the I. G. Farbenindustrie A.-G., in German patent 564,018, issued November 12, 1932, applied for July 5, 1931, claim a product for combating flies and similar insects consisting of urethanes or their substitution products together with extracts from pyrethrum, or Derris root, or synthetic insecticides.

Eichorn (24), in German patent 630,483, issued May 28, 1936, applied for November 22, 1932, claims the process of manufacturing an insecticide containing derris extract which consists in extracting finely divided Derris root with a dilute solution of sodium bisulphite at 40° to 50° C., neutralizing the solution and concentrating in vacuo.

Examples are:

(1) 100 parts ground or shredded derris root and 100 parts of a 10 percent sodium bisulphite solution are ground 3 to 4 hours in a mill at 40° to 50° C. The solution is filtered, neutralized and evaporated in vacuo.

(2) 100 parts derris root are extracted in a percolator with 200 parts of a 10 percent sodium bisulphite solution, with recirculation, and the extract treated as in Example 1.

(3) The extraction of the derris root is carried out in a diffusion battery.

The extract obtained from derris by extraction with water amounts to 6 percent of the weight of the root and contains 4.23 percent rotenone, whereas the extract obtained with sodium bisulphite amounts to 10 percent of the weight of the root and contains 16.95 percent rotenone.

Tests on insects gave the following results:

	Test animal	Result	
		dead Percent	stunned Percent
1. Derris powder*with water as paste (10:50)	Cockroaches	50	50
2. Aqueous extract of derris root (1:50)	"	-	100
3. Sodium bisulfite extract of derris root (1:50)	"	60	40
4. Aqueous extract of derris root (1:50)	Japanese green-house locusts	80	20
5. Sodium bisulfite extract of derris root (1:50)	"	100	
6. Aqueous extract of derris root (1:50)	Mole crickets	Stunned after 25 min. dead after 60 min.	
7. Sodium bisulfite extract of derris root (1:50)	-	Stunned after 17 min. dead after 23 min.	

* contained 2 percent rotenone.

Chronok (15), in German patent 643,804, issued April 17, 1937, applied for February 7, 1932, claims an insecticide consisting of an extract of a rotenone-bearing root in aliphatic hydrocarbons, especially petroleum, hexachloroethane, ammonium linoleate, and water. An example is dorris powder 7 parts, hexachloroethane 12 parts, ammonium linoleate 4 parts, petroleum 25 parts, methyl salicylate 2 parts, and water 50 parts.

The Booth Steamship Co. Ltd. and Ward (9), in German patent 646,570, issued June 17, 1937, applied for September 11, 1935, in Great Britain March 30, 1935, make essentially the same claims as in British patent 437,171 and United States patent 2,056,438.

Japanese Patents:

Iiba and Makatura (42), in Japanese patent 31,478, issued May 20, 1931, claim a mixture of 67.6 percent trichloroethane and 32.4 percent methyl alcohol (the minimum boiling mixture) for use as a solvent in extracting pyrethrum flowers or dorris root. The solvent is distilled and the residue used as an insecticide.

Imai (43), in Japanese patent 31,645, issued June 3, 1931, patented an insecticide consisting of vegetable volatile oils (such as turpentine or camphor oils), naphthalene, and extracts of dorris roots.

Isihara (47), in Japanese patent 31,875, issued June 20, 1931, patented a process in which dried roots of plants belonging to the dorris group are cut into pieces and powdered under cooling by a special stamping machine. The powder passed through a 60-mesh sieve is used as an insecticide.

Johore Patent:

A report from Boehringer (6) [Japanese Firm Claims New Process of Obtaining Toxic Substance from Dorris], Assistant Trade Commissioner, Singapore, dated March 8, 1934, stated that the Nottai Sangyo Kabushiki Kaisha, of Tokyo, Japan, has been granted the exclusive privilege of making, selling and using, in the State of Johore, an invention for "A new or improved process of obtaining insecticidal or other toxic substances from plants of the dorris species." This privilege is for the term of 16 years from June 19, 1933.

Swedish Patents:

Wikström (103), in Swedish patent 86,083, issued April 15, 1936, applied for March 16, 1934, claims as an insecticide cyndol [cymene] or its derivatives. Dissolved or suspended in cyndol may be pyrethrum, dorris, quassia, rotenone, etc.

Cynol can be used to extract the active principles of pyrethrum, derris, and quassia.

Other valuable components that can be used are chlorinated or sulfonated ethereal oils. These ethereal oils act as contact poisons. Some examples follow:

I.	Derris cynol extract (15 percent)	10 parts
	Adhesive	1 part
	Water	1,000 parts
II.	Pyrethrum cynol extract (15 percent)	1 part
	Derris Cynol extract	1 part
	Water	200 parts
III.	Pyrethrum cynol extract (15 percent)	1 part
	Derris cynol extract	1 part
	Quassia cynol extract	1 part
	water	300 parts
IV.	Pyrethrum cynol extract (15 percent)	1-60 parts
	Derris or	
	Quassia cynol extract	1-60 parts
	Chlorinated or sulphonated ethereal oils	2-60 parts
	Soaps or emulsifying agents	200-400 parts
	Water	2,000-200,000 parts

A common mixture consists of

Pyrethrum cynol extract (15 per-	} 15 parts	} Diluted with
cent) or Derris - or quassia cynol		
extract; Chlorinated or sulfonated		
ethereal oils	6 parts	.25 to 15
Soaps	79 parts	percent solns

Wikström (104), in Swedish patent 87,625, published October 13, 1936, applied for March 16, 1934, claims an insect killing material characterized as follows: That it contains cynol or a derivative thereof as carrier or solvent for one or several chlorinated or sulfonated ethereal oils--but not such insect or pest killing materials or poisons as extracts of pyrethrum, derris, quassia, or the active derivatives therefrom.

Swiss Patent:

The F. Hoffmann-LaRoche and Co. (37), in Swiss patent 124,290, issued January 16, 1928, applied for October 5, 1926, claim an insecticide

containing the dried, pulverized root of Derris elliptica or the crude or pure extract therefrom in an emulsified form. The following examples are given: (1) One part crude ether extract of the roots of D. elliptica and 2.5 parts of a 10 percent ether solution of ozonized rape oil (obtained by treating 10 parts rape oil in 60 volume parts chloroform with ozone for 4 hours, distilling off the chloroform, taking up the residue in aqueous alcohol, and shaking the solution with ether) are dissolved in 30 parts alcohol, then treated with 10 parts ether, and, to facilitate the production of an emulsion, normal sodium hydroxide is added until a reddish color is obtained by phenolphthalein. This solution is slowly and with constant stirring dropped into 60 parts distilled water at 70° to 80° C. Within certain limits the solution may be concentrated or diluted without the separation of the emulsified products. (2) One part crystallized rotenone, that is, the purified ether soluble principle of the roots of D. elliptica, together with 0.1 to 0.2 part cholesterinozonide, is dissolved in 20 parts acetone and 10 parts ether and treated with 10 parts alcohol, and, in order to facilitate emulsification, sodium hydroxide is added until faintly alkaline. The solution is dropped into 60 parts water at 70° to 80° C. with vigorous stirring. (3) One part rotenone and 0.1 part phytosterinoxide are dissolved in 12 parts alcohol and 4 parts ether and, to facilitate emulsification, made slightly alkaline with sodium hydroxide solution. The solution is slowly dropped with stirring into 50 parts water at 70° to 80° C. (4) One part finely pulverized roots of D. elliptica and 0.1 part ozonized cholesterol are treated with hot alcohol, then with 10 parts ether, and the residue is filtered off. To facilitate emulsification sodium hydroxide solution is added until the reaction is faintly alkaline. The solution is slowly dropped into 60 parts distilled water at 70° to 80° C., with vigorous stirring. Claim is made for an insecticide consisting of oxidation products of unsaturated fatty bodies and the insecticidal constituents of the root of Derris elliptica in emulsified form.

United States Patents:

McDougall, McDougall, and McDougall (66), in United States patent 1,035,156, issued June 17, 1913, applied for September 8, 1911, claim a composition for use as an ingredient for animal washes, insecticides, and vermifuges, comprising a concentrated extract of trees of the Derris family, said extract giving a red color when treated with concentrated sulphuric and nitric acids and a fugitive brilliant green color on the further addition of concentrated ammonia solution, as described. The powdered dried roots are extracted with a suitable solvent, such as benzine, and the extract is evaporated to a gummy mass. A suitable extraction apparatus is shown - diagrammatically. For a sheep dip a mixture of 5 ounces of the compound and 1 pound of soft soap may be used, such mixture being adapted for use with 100 gallons of water; or 5 ounces of the compound may be mixed with 15 ounces of soft soap, the mixture then being absorbed into a powder by means of sulphur to make a total weight of 10 pounds. This preparation should be used with 100 gallons of water. During the manufacture of the dip,

wash, or insecticide, a suitable water-softening reagent or reagents may be added to the composition. In like manner a suitable antiseptic or bactericide and also a coloring matter may be added.

Dow and Hale (22), assignors to the Dow Chemical Co., in United States patent 1,514,377, issued November 4, 1924, applied for March 31, 1920, claim an insecticide consisting of the halohydrin extract of dorris admixed with benzyl alcohol. They also claim an insecticide comprising a solution of benzyl alcohol and a halohydrin, the amount of the latter being sufficient to render such alcohol freely soluble in water. Ethylene chlorohydrin or propylene chlorohydrin are suitable halchydrius. The preferred proportions are 1 part alcohol to 1 part chlorohydrin.

"We have also found that the combination of ingredients just described serves as an excellent solvent for various so-called deadly insecticides, such as timboin, nicotine and dorris (or "Fish poison"). In utilizing such combination of ingredients in this connection, we preferably extract the poison first with the halohydrin, and admix the extract with benzyl alcohol in desired proportions, depending upon specific use to be made of insecticide. Furthermore, such combination of ingredients may readily be mixed with inert dry materials, such as lime, lead, calcium or magnesium arsenate, and the like, capable of absorbing the same, and can then be applied in the form of a powder or dust, with highly satisfactory results."

Bagnall-Bull (5), in United States patent 1,522,041, issued January 6, 1925, applied for July 22, 1924, makes the same claims as in British patent 214,822.

Fryer (27), assignor to McDougall and Yalding, Ltd., in United States patent 1,583,681, issued May 4, 1926, applied for November 29, 1924, claims an insecticide comprising dorris, a vehicle of a sulphonated fatty body, and a colloidal addition agent. The preferred insecticide consists of 1 part of dorris extract to 10 to 50 parts of sulphonated oil or wax, for example, sulphonated castor oil. If it is desired to use this material in combination with lime-sulphur so as to produce a combined insecticide and fungicide, precipitation of the material may be prevented by the addition of a colloidal agent, of which casein (5 to 20 percent) is found most suitable. Powdered casein may be dissolved in warm sulphonated oil.

The active principles of dorris are extracted with benzene or other suitable volatile solvent. An extract of perkakal may be similarly used. The sulphonated oil may be sulphonated sperm oil. This insecticide may be used alone or in admixture with lead arsenate and bordeaux mixture.

Dennis (17), on April 16, 1923 filed application for United States patent 1,621,240, which was granted him on March 15, 1927. This patent covers the use as an insecticide and vernifuge of ground cube root with

the fibrous element removed. Cube is stated to be a woody shrub found in Peru and other South American countries which is classified by some native botanists as of the family Solanaceae. Directions are given for preparing a solid extract of cube by grinding the roots or plants, extracting with a suitable solvent such as benzol, or alcohol, filtering, and evaporating. The solid extract has a light straw color and is odorless and tasteless.

"For identification, the powder may be subjected to the action of concentrated sulphuric acid and dilute nitric acid, whereupon it gives a bright clear red color. By the addition of potassium hydroxide in nearly equal volume, a vigorous action is obtained, and the solution turns to a yellowish pink. A further addition of potassium hydroxide produces a stable brown solution.

"The compound in the form of powder or dust may be used as an insecticide with talcum powder, cornstarch or other carrier. It is very effective against flies, roaches, and other insects. It is my belief that the powder sets up an irritation of the breathing apparatus which results in early death.

"The compound can be used with water or with soap and water or other emulsifying agent, as a wash or spray for plants, animals, or trees. It is effective against aphids, beetles, some insects, and worms.

"It can be used as an extract with alcohol for similar purposes, for instance as a wash for ticks on cattle.

"As an alcoholic extract, tests show the solution to have slightly greater effectiveness when sprayed on the cotton aphid than nicotine sulphate. Experience shows that it is eight times as effective as similar derris extract.

"The proportions of the compound and the carrier may be varied as desired for different purposes.

"For certain purposes, it is desirable to use the alcoholic extract in a soap and water emulsification. A suitable solution for aphids can be made by using the extract from one-third pound of powder in one hundred gallons of soap solution."

In a reissue of this patent (reissue 12,667 of November 22, 1932, applied for February 25, 1929) Dennis (18) corrected the information concerning the botanical origin of cube given in his original patent 1,621,240, stating: "While the exact botanical classification of the plant is not altogether certain it is believed to be a species of the genus Lonchocarpus of the family Leguminosae."

Takei (28), assignor to Zaidan Hojin Rikagaku Kenkyujo, in United States patent 1,724,626, issued August 13, 1929, applied for August 31,

1925, in Japan, September 8, 1924, claims the process of producing a liquid insecticide containing the effective ingredient of the derris species which comprises extracting raw material containing the aforesaid ingredient with a volatile organic solvent therefor, mixing the extract with an anhydrous alkali-free organic preservative for the ingredient, evaporating the solvent from the mixture and mixing the resulting product with an organic colloidal material in an aqueous medium. Examples are: (1) A solution of 10 liters of ether added to 10 grams of rotenone, is mixed with a mixture of 400 grams of fish oil with 40 grams of hard oil. After sufficient agitation, all or the greater part of the ether is recovered by evaporation and condensation and thereby a semi-solid mixture is obtained as a primary material. Five hundred grams of soap are mixed with 100 kilograms of water to prepare a colloidal aqueous solution as a secondary material. In use, the primary reagent is mixed with the colloidal aqueous solution and it may be suitably diluted with addition of 100 kilograms of water when used as insecticide. The colloidal solution or secondary reagent may be prepared at any time by the users. (2) Ten liters of ether are added to 1 kilogram of the derris root. After 4 days, the extract is added to 400 grams of chrysalis oil and 100 grams of lard. After evaporating the ether, a semi-solid substance is obtained as a primary reagent. The preparation of the secondary reagent is same to that of example 1. (3) Twenty liters of petroleum ether are added to 1 kilogram of derris root. After 4 days, the extract is added to 200 grams of beeswax and 50 grams of pine resin. After solution, the larger portion of the petroleum ether is recovered by evaporation and condensation and thereby the semi-colloidal material is obtained as the primary material or reagent. The secondary reagent may be prepared by adding a colloidal solution in which 200 grams of soap are dissolved in 100 kilograms of water.

Takei attributes the poor qualities of rotenone insecticides on the market to the combined action of alkali and water. A superior product is made by mixing rotenone with one or more kinds of oils, fats, waxes, resins, higher hydrocarbons, higher alcohols, higher fatty-acids which are entirely free from water and alkali to form a stable preservative mixture and this mixture is mixed with colloidal aqueous solution such as soap water in use.

Rotenone may be extracted with such solvents as methyl alcohol, ethyl alcohol, ether, petroleum ether, chloroform, or carbon tetrachloride. The oils mean all kinds of animal oils, such as sardine oil, herring oil, cod-liver oil, shark oil, chrysalis oil, or a vegetable oil, such as coconut oil, bean oil, rapeseed oil, and castor oil. The fatty oil may be a material such as lard, beef tallow, animal hard oil, and vegetable hard oil. Waxes, such as wood wax, and beeswax may be used, also resins, such as pine resin, and copal. Higher alcohol includes glycerin, and higher hydrocarbon means paraffin, etc.

Colloidal aqueous solution includes solution of soap, glue, egg-albumin, bean milk, dextrine, starch, bengal isinglass, animal albumin, etc. Sometimes to said aqueous solution is added a little quantity of methyl alcohol, ethyl alcohol, chloroform, ether, camphor oil, lemon oil, and other solvents.

Otori and Nakazawa (78), assignors to Zaidan Hojin Rikagaku Kenkyujo, in United States patent 1,745,907, issued February 4, 1930, applied for February 10, 1928, in Japan, February 18, 1927, claim a process for producing a liquid insecticide containing the effective ingredient of derris species, which comprises forming a primary material in which the effective ingredient is mixed with an anhydrous and alkali-free material of an oily nature and dissolved in an anhydrous solvent such as benzol, carbon tetrachloride, or trichloroethane, and then mixing the primary material with a soap solution to form a colloidal solution. Examples are: (1) To 100 grams of powdered derris root, 10 liters of ether are added to extract the effective ingredient of the derris root. After extraction is complete, the whole or greater part of the ether is evaporated and recovered. To the residual effective ingredient a mixture of 150 grams of anhydrous and alkali-free fish oil and 150 grams of benzol (as the anhydrous solvent) are added, so that a solution of the effective ingredient in benzol results. This is the improved primary material. The soap solution is easily prepared by, for instance, adding 500 grams of soap to 100 kilograms of water. The soap solution is mixed with the improved primary material before use. (2) To 1 kilogram of powdered derris root, 5 kilograms of benzol are added and the effective ingredient of the derris root is extracted while warming the benzol. The benzol is recovered by evaporation. To the residual effective ingredient, a mixture of 600 grams of anhydrous and alkali-free fish oil and 600 grams of carbon tetrachloride (as the anhydrous solvent) are added to form a solution of the effective ingredient. This is the improved primary material. The soap solution may be the same as exemplified in Example 1. Trichloroethane as the anhydrous solvent can be used as well. Also in the two examples given, the improved primary material can be easily prepared in the form of powder when 800 grams of anhydrous gum arabic are added.

O'Kane (79), in United States patent 1,786,125, issued December 23, 1930, applied for May 18, 1927, claims a fungicide and insecticide consisting of a hydrocarbon white oil emulsion carrying a colloidal copper containing material. If desired this refined oil may be used to extract any suitable organic material such as pyrethrum of various species, derris of various species, or other substances that will furnish an additional killing agent effective against insects. Or, such materials may be added to the refined oil. The colloidal metal derivative may be, for example, colloidal cuprous oxide.

Sanders (84), in United States patent 1,803,870, issued May 5, 1931, applied for October 3, 1927, claims a process which comprises forming a colloidal suspension containing a metal, the salts of which are adapted to kill low forms of life, by reacting upon a compound of such a metal with a casein compound, the proportion of the casein compound being not less than 1 pound of actual casein to 2 pounds of the metallic metal.

"In making up a paste copper fungicide it is preferable to add 4 lbs. of casein or 10 lbs. of calcium caseinate to 50 lbs. of paste formed by adding 25 lbs. of calcium hydroxide and 25 lbs. of copper sulphate to 50 gals. of water and filtering, as otherwise

some of the colloidal copper would be lost in the filter water. It will be understood, furthermore, that the above mixtures may be added to other insecticides or fungicides, either before or after causing them to react in water. For example, dry arsenical compounds or derris or pyrethrum or other insecticidal powders may be added to the dry mixture of the copper compound and the casein compound, or various fungicides or insecticides, such as emulsified oils, nicotine or derris compounds, etc., may be mixed with the wet colloidal copper mixture."

Lingler (57), in United States patent 1,820,394, issued August 25, 1931, applied for March 9, 1929, in Germany, March 12, 1928, assigned to the Deutsche Gesellschaft für Schädlingbekämpfung M.B.H. of Frankfurt-on-the-Main, Germany, claims a method of fumigating which consists in converting a cyanide of a metal selected from the group comprising alkaline earth metals and magnesium, into shaped bodies of a size the hydrocyanic acid content of which corresponds to the amount required for fumigating, reducing the shaped bodies into powder, and blowing the said powder into the space to be fumigated.

"According to one method of carrying my invention into effect other auxiliary substances, for example, such as are also capable of acting upon the vermin to be destroyed, as for example, nicotine or suitable nicotine compounds, as for example, nicotine sulphate, pyrethrum, derris root or stimulating or preventative substances, which volatilize simultaneously with the hydrocyanic acid which is given off may be incorporated with the pulverous alkaline earth cyanide compounds or the shaped bodies to be produced therefrom."

Sanders (85), in United States patent 1,847,540, issued March 1, 1932, applied for March 11, 1930, claims a method of coating particles of insecticide dust and causing them to become floatable on water by utilizing the heat of slaking lime for melting stearic acid to coat the particles. Sufficient water is added to quicklime to convert all or a portion of it to hydrated lime.

"This water may carry in suspension or in solution a poison or the poison may be added separately either before or after the water. While the mass is hot and being agitated I add a fatty acid enough to coat most of the particles in the mass and render them floatable. The fatty acid may be mixed with either the lime or the poison either before or after adding the water to the lime without departing from the spirit of the invention. I then grind and air float in a suitable mill. An example of the proportions used is: 300 lbs. quicklime, 300 lbs. paste copper arsenite. When at its maximum heat and under agitation add 12 lbs. of stearic acid. This mixture should be ground before using to get the best results but may be used directly." "Other insecticides other than arsenic may be used as finely ground pyrethrum, hellebore, derris, nicotine dust, etc."

McGill (72), in United States patent 1,854,948, issued April 19, 1932, applied for November 7, 1927, claims a solution containing rotenone adapted for use in the mothproofing of fabrics. He also claims a mothproofing solution containing an extract of derris, or of haiari.

"The invention will be readily understood from the following example.

"One pound of derris root is ground to a powder and admixed with one gallon of petroleum naphtha, boiling between approximately 300 and 400° F. The mixture is allowed to stand at room temperature for a considerable period, for example, five to seven days, being occasionally agitated during this period. The hydrocarbon oil containing the desired active principles of the derris root is then removed, filtered clear and, if desired, minute amounts of essential oils or other suitable odoriferous substances may be added.

"In preparing the extract, if it is desired to hasten the operation, the mixture of oil and powdered root may be heated mildly, say to a temperature of 150 to 200° F., and the period required for the extraction of the active principles by the oil is thereby substantially reduced, say to 24 or 36 hours.

"Extracts of haiari may be produced in the same manner. In using black haiari, it is preferred to extract both the roots and stem, while in the case of white haiari, it is preferred to extract the stem and leaves.

"The solution produced as described is particularly suitable for use by dipping the fabrics therein, pressing out excess solution and drying. In this way, rotenone is distributed over every fiber of the fabric, which is thereby rendered moth proof for a considerable time. The moth proofing dip may suitably be the last step in a dry cleaning operation.

"Where it is desired to apply the active principle by means of a spray, it is preferred to employ a higher boiling naphtha cut, for example, one boiling between 450-550° F., since the slower evaporation of such naphtha permits a more complete penetration of the extracts into every portion of the fabric or garment which might not be directly exposed to the spray.

"While it is preferred to employ petroleum naphthas as the extracting fluid, it must be understood that the invention is not intended to be limited in this respect since alcohol, benzol, ether, and other similar organic solvents are also effective in extracting and holding in solution the active principles of derris root and other substances containing rotenone.

"Rotenone has important properties which render it invaluable as a mothproofing agent. It can be applied with great ease. It has no pronounced or objectionable odor. It is of relatively low volatility so that it persists in the fabrics for a considerable time. It is highly toxic and repellent to the moth larvae, but is non-injurious to human beings and warm blooded animals."

Jones (49), in United States patent 1,928,256, issued September 26, 1933, applied for August 23, 1930, claims an insecticide comprising water, rotenone colloiddally dispersed therein, and a small proportion of a pyridine base. For example, 1 gram of pure rotenone is dissolved in 100 cc. of pyridine. One cc. of this solution is added to 100 cc. of distilled water and shaken gently. A pale opalescent colloidal solution results which shows no evidence of separation over an extended period of time. The various methyl pyridines (alpha, beta, and gamma picolines), the dimethyl, trimethyl, ethyl, allyl, and other alkyl-pyridines may be employed in place of pyridine. Similarly the dipyridyls and other compounds containing a pyridine nucleus may be used. These compounds are included under the term "pyridine base". In place of pure rotenone, a crude rotenone, or an extract of a plant containing rotenone may be used. For example extractives from Derris elliptica, Lonchocarpus, and other plants known to contain rotenone may be employed.

Jones (50), in United States patent 1,928,968, issued October 3, 1933, applied for August 23, 1930, claims a process of producing a liquid insecticide containing rotenone in a colloidal state of dispersion which consists in dissolving rotenone in acetone, and adding this solution to water in the presence of tannic acid with subsequent mixing. For example, 4 grams of pure rotenone and 2 grams of tannic acid U. S. P. are dissolved in 100 cc. of acetone, a clear solution resulting. Two and one-half cc. of this solution is added to 1,000 cc. of distilled water and shaken gently. An opalescent colloidal solution results which shows no evidence of separation during an extended period of time. In attempts to form suspensions such solvents as acetic acid, acetone, diacetone alcohol, ethyl alcohol, ethyl formate, ethylene chlorohydrin, and pyridine have been tried. With the exception of pyridine, none of these gave satisfactory suspensions when no protective colloid was added. Acetic acid, ethyl alcohol, ethyl formate, ethylene chlorohydrin, and pyridine did not give permanent dispersions in the presence of a protective material such as tannic acid. Acetone and diacetone alcohol, on the other hand, have been found to give very satisfactory results when used in the presence of the proper amount of suitable protective material.

Experiments have been made to test the efficacy of a number of protective colloids for this purpose, the protective material being dissolved or suspended in water and an acetone solution of rotenone added. Among these were gum acacia, gum karaya, gum ghatti, gelatin, starch, agar-agar, saponin, tannic acid, triethanolamine oleate, ammonium alginate, potassium oleate, calcium caseinate, diatomaceous earth, bentonite, and lithopone. Tannic acid was found to be the most suitable material as regards suspending or protecting power, as well as from the standpoints of solubility and cost.

In place of acetone methyl ethyl ketone, diethyl ketone, methyl ethyl acetone and acetyl acetone may be used. In like manner such aryl compounds as phenyl ethyl ketone and o- or m-phenyl tolyl ketone may be employed in place of acetone or di-acetone alcohol. In place of pure rotenone, a crude rotenone, or an extract of a plant containing rotenone may be used. For example, extractives from Derris elliptica, Lonchocarpus and other plants known to contain rotenone may be employed.

Grant (32), in United States patent 1,934,057, issued November 7, 1933, applied for October 31, 1928, assigned to the Standard Oil Development Company, claims an insecticide or insect repellent comprising a petroleum white oil, derris extract, and an emulsifying agent. The derris roots are extracted with a volatile solvent, such as benzol, isopropyl alcohol, etc. The extraction may be carried out by any of the known methods, such as percolation, maceration, etc. This method is especially adapted to prepare concentrated extracts and consists of causing the solvent to flow over and through the substance to be extracted without flooding it. The volatile solvent is then evaporated partially or wholly, preferably in vacuo, leaving a concentrated derris extract. The latter is then emulsified with a petroleum oil, preferably of the kind known as white oil, and made by intensive acid treatment of gas-oil or lubricating-oil fractions, such as machine oil, transformer oil, light or medium lubricating oils, etc. The emulsification may be carried out by dissolving an emulsifying agent, such as soap, in the white oil and feeding it, together with the dry derris extract, into a colloid mill to obtain a stable emulsion. Some water may be thoroughly admixed with this product before marketing.

Another method which may often be used with advantage is the following. The insecticidal or active principles of the derris root are extracted directly with the "white oil." Volatile solvents such as alcohols; ethers; esters; ketones, for instance acetone; aromatic hydrocarbons, for instance benzene; or chlorinated hydrocarbons may be added to the white oil to increase its solvent power. In this case, however, it is preferable to distill off subsequently most of the volatile solvents from the product obtained. A similar result may be obtained by adding steam distilled pine oil, terpineol, phenol, cresol, or their homologs, cyclohexanol and its homologs hydrogenated naphthalenes such as tetralin or decalin, chlorinated naphthalenes such as halowax oil, etc., to the white oil to increase its solvent power for the derris extractives. The ingredients just mentioned cannot and should not be removed by subsequent distillation but remain in the product and confer upon it a fungicidal and bactericidal effect in addition to its insecticidal properties and facilitate its emulsification. The derris-white oil extract is then mixed with an emulsifying agent, such as fatty soap, rosin soap, saponin, soaps of petroleum sulphonic acids, soaps of sulfonated vegetable or animal oils, to form a composition capable of dispersing in water. Any one soap or a mixture of several soaps may be used. Part of the soap may be added to the white oil before the extraction because it has been found that the presence of a soap facilitates the extraction. Oil-soluble sulphonates are preferably used as an emulsifying agent, and are obtained in the refining of said white oils.

The product may be marketed either in the form of a homogeneous liquid mixture of oil, derris extractives, emulsifier and added solvents or fungicides, practically free from water, or in the form of a stock emulsion of creamy or buttery consistency containing up to 35 percent of water. Its composition may greatly vary according to the requirements of the market and the transportation facilities. Products containing as high as 75 percent derris extractives may be prepared. The practical limits of the components are the following:

1 to 25 percent derris extractives, 40-80 percent white oil, 5-25 percent soap or other emulsifier and fungicide, 0-35 percent water. Usually the percentage of the derris extractives is kept within the limits of 1 to 10 percent.

The marketed product is further diluted with water before use, to such extent that the composition of the final diluted spray is about the following: 0.06 to 0.25 percent derris extractives, 0.5-1.00 percent petroleum white oil, 0.2-1.00 percent soap, 97-99.00 percent water.

This new insecticide is effective against pests such as the various aphids, tent caterpillars, scale insects, redmite, tomato moth, cabbage worm, sawflies, etc.

An example of preparing this insecticide is as follows:

Two kilograms of ground derris roots were extracted with 4 liters of benzol. Distilling off the solvent in vacuo, 113 grams of dry extract were obtained, representing 5.65 percent of the dry derris roots, and having the appearance of a reddish-brown resin. Eighty grams of this extract were fed into the colloid mill together with a solution of 750 grams of oil soluble sodium solphonate in 2.5 liters of "Wyrol", a commercial brand of white oil. Water was also added in the amount of 1.5 liters. A thick emulsion resulted similar in appearance to mayonnaise and ready for marketing. Part of this preparation was diluted with 50 times its weight of water and gave a final spray which was effective when sprayed on trees as an insecticide.

Badertscher (2), assignor to McCormick & Co., Inc., in United States patent 1,938,652, issued December 12, 1933, applied for August 20, 1931, claims a horticultural spray including pine oil, an insecticide, and dipentene, the dipentene being present in amount in excess of the amount normally present in pine oil and in quantity sufficient to substantially negative the tendency of the pine oil to burn foliage. Any suitable insecticide may be included as, for example, nicotine or nicotine sulphate, rotenone, pyrethrins as an extract of pyrethrum flowers, quassia as an extract of quassia chips, etc.

Grant (33), assignor to Standard Oil Development Co., in United States patent 1,940,646, issued December 19, 1933, applied for October 19, 1927, claims in the manufacture of insecticides, the improvement which comprises extracting the active insecticidal principles from vegetable matter of the group consisting of pyrethrum, derris, cube, and tephrosia, with a

mixture of an organic solvent, a blending agent and an emulsifying agent. Preferred organic solvents are naphtha, kerosene, and light lubricating oil. Suitable blending agents are: oil soluble sulphonates, Turkey red oil, rosin soap, sulphonated vegetable or mineral oils, steam distilled pine oil, terpinol, cresol, cyclohexanol, dichlorobenzene, chloronaphthalene, one or more of the higher alcohols, such as isopropyl or secondary butyl or secondary amyl alcohol, ketones, or cyclohexanol or its homologues. Some of the above materials may serve both as blending agents and as emulsifying agents. Such materials having this dual property are oil soluble sulphonates, Turkey red oil, sulphonated vegetable or animal oils and to a certain extent also rosin soap. The active principles may be extracted with other solvents, for example, alcohols (ethyl, isopropyl, etc.), benzol, or carbon tetrachloride. The extract so prepared may be concentrated to yield an oleo-resin which may be mixed with oil-soluble sulphonate in suitable percentages. Various substances may be added to the composition either before or after the organic material is extracted, and before or after concentration by evaporation or distillation. Phenols, cresols, pine oil, etc., are suitable for this purpose. Illustrative composite solvents are: (1) kerosene 31.6 cc, oil-soluble sulphonate 18.4 cc, water 2.0 cc; (2) kerosene 31.6 cc, oil-soluble sulphonate 13.4 cc, secondary butyl alcohol 10.0 cc, water 68.0 cc; (3) kerosene 31.0 cc, oil-soluble sulphonate 18.0 cc, ethyl methyl ketone 10.0 cc, water 35.0 cc; (4) petroleum white oil (vis. 75-85 sec. Saybolt at 100° F.) 45.0 cc, oil-soluble sulphonate 5.0 cc, dichlorobenzene (crude) 9.5 cc, water 2.0 cc; (5) mineral seal oil 171.3 cc, oil-soluble sulphonate 30.2 cc, potash rosin soap 10.0 g., hexahydrophenol 10.0 cc, ethyl methyl ketone 10.0 cc, cresol, U.S.P., 10.0 cc, water 24.0 cc; (6) petroleum white oil (vis. 75-85 sec. Saybolt at 100° F.) 128.0 cc, oil-soluble sulphonate 14.2 cc, secondary amyl alcohol 6.6 cc, steam distilled pine oil 6.6 cc, chloronaphthalenes (crude) 6.6 cc, potash rosin soap 8.6 gm., water 17.5 cc.

Badertscher (3), assignor to McCormick & Co., Inc., in United States patent 1,940,899, issued December 26, 1933, applied for January 21, 1932, claims the method of producing a horticultural dust which includes spraying a non-alkaline inert material in finely divided form with the active principle of pyrethrum in solution in a volatile solvent while agitating the inert material and maintaining it at a temperature at which the solvent will evaporate.

The non-alkaline inert material may be a siliceous earth, talc, bentonite, etc. The active principle of pyrethrum may be used in admixture with rotenone. A horticultural dust made in accordance with this invention may comprise 80-95 inert material and 5-20 pyrethrum, or pyrethrum and rotenone, and the coated, impregnated, inert material may, if desired, be admixed with ground derris root or ground cube root in the proportion of 60 parts inert material to 40 parts of ground derris or cube root, or a mixture thereof.

Jones (51), in United States patent 1,942,104, issued January 2, 1934, applied for February 20, 1933, claims a process for making a chemical compound of rotenone and carbon tetrachloride which consists substantially in extracting the roots of plants of the genus Desmodium (Derris) with warm carbon tetrachloride, and crystallizing. Roots of Lonchocarpus or Spatholobus may be used likewise.

For example 50 grams of the roots of Dequelia (Derris) sp. (tuba root) is completely extracted in a continuous extractor with carbon tetrachloride. The extract is evaporated to a volume of 25 cc. and cooled in a refrigerator. When crystallization is complete the separated material is filtered, excess solvent removed by suction and the needlelike crystalline product dried in the air. The solvate $C_{23}H_{22}O_6 \cdot CCl_4$ contains 71.9 percent by weight of rotenone and has highly valuable properties as an insecticide, and mothproofing agent.

Mills and Fayerweather (75), assignors to the Dow Chemical Co. in United States patent 1,942,827, issued January 9, 1934, applied for March 27, 1933, claim 1,2-dihydroxy-4-tertiary alkyl benzene, especially the tertiary butyl and tertiary amyl derivatives. As little as between 0.001 and 0.05 percent of these compounds in gasoline inhibits gum formation. These new compounds may also be employed as stabilizers for insecticide compositions, such as pyrethrum and rotenone. They may also be used as antioxidants in vegetable and animal fats, waxes, soaps, etc.

Haller and Schaffer (34), in U. S. patent 1,945,312, issued January 30, 1934, applied for April 10, 1933, claim the process of making di-hydrorotenone which comprises hydrogenating a rotenone-bearing plant extract dissolved in an organic solvent in the presence of a nickel catalyst obtained by alloying equal parts of nickel and aluminum and then dissolving out the latter with aqueous sodium hydroxide. Benzene, acetone, ethyl acetate, and ether are suitable solvents. The reaction proceeds at atmospheric pressure and at room temperature and the yield of dihydrorotenone is about 90 percent of the theoretical.

For example 50 grams of the ground root of Dequelia (Derris) sp. (tuba root) is exhaustively extracted in a continuous extractor with acetone. The extract is concentrated to 50-75 cc. and placed in the reaction bottle of a suitable catalytic reduction apparatus and 3 grams of nickel catalyst suspended in 10 cc. ethyl alcohol are added. The apparatus is evacuated, then filled with hydrogen and the mixture is shaken with hydrogen until the rate of absorption of hydrogen is very slow. The solution is decanted from the catalyst, filtered, and concentrated to a small volume, and 10-25 cc. of ethyl alcohol are added. After standing for a short time the crystallization of dihydrorotenone is usually complete. The product is practically pure dihydrorotenone.

Bousquet and Tisdale (11), assignors to E. I. du Pont de Nemours & Co., in United States patent 1,954,517, issued April 10, 1934, applied for July 8, 1932, claim the method of killing insects which comprises contacting the insects with a water emulsion of beta beta-dichlordiethyl ether and an insecticide of the group consisting of water-insoluble dithiocarbonates, water-insoluble thiuram sulphides (especially tetraethyl thiuram monosulphide) and the toxic ingredient of derris root.

Examples are:

Nasturtium plants infested with black aphids were sprayed with a soap emulsion containing 0.1 percent of a solution composed of 10 percent of the toxic ingredient from derris root and 90 percent beta beta-dichlordiethyl ether. After 24 hours, more than 99 percent of the aphids were dead.

Common houseflies were sprayed with kerosene containing 2 percent of a beta beta-dichlordiethyl ether extract of derris root. Approximately 90 percent of the flies were paralyzed in 6 minutes and 90 percent were dead in 24 hours.

Dougherty and Anderson (21), in United States patent 1,963,838, issued June 19, 1934, applied for May 23, 1933, described a portable sprayer which uses steam generated by an electric heating unit to expel insecticidal fluids. Rotenone is mentioned as a botanical derivative which can be used in such an insecticidal composition.

Fulton (28), assignor to Stanco. Inc., in United States patent 1,967,024, issued July 17, 1934, applied for June 6, 1929, claims an insecticide comprising pyrethrins and the active extract containing rotenone obtained from a plant. For example pyrethrum extract is prepared by extracting pyrethrum flowers with 8 times their weight of kerosene and decolorizing the obtained solution with 3 percent of its weight of chemically prepared absorptive clay, such as "Filtrol". This is improved by dissolving 0.025 percent by weight of rotenone in the extract. In case it is desired to dissolve a higher percentage of rotenone it is necessary to use a mixed solvent. If for instance 1 percent by weight of chloroform is added to the pyrethrum-kerosene extract 0.1 percent by weight of rotenone is soluble in it, i.e., four times as much as in the absence of chloroform. A benzol extract of derris may be used instead of pure rotenone. Carbon tetrachloride is also mentioned as a solvent.

Bousquet (10), assignor to the Grosselli Chemical Co., in United States patent 2,006,227, issued June 25, 1935, applied for October 19, 1932, claims a contact insecticide comprising an aqueous preparation containing technical soya bean lecithin as the essential active ingredient and sulphonated fish oil or butyl alcohol or other ingredients as a dispersing agent. It is stated lecithin preparations are compatible with other contact insecticides such as nicotine, rotenone, and pyrethrum preparations.

James (48), assignor to C. P. Byrnes, Trustee, in United States patent 2,006,456, issued July 2, 1935, applied for March 12, 1930, claims in the process of treating sulphonated products obtained by treating lighter petroleum fractions such as gasoline with sulphuric acid to obtain an emulsifiable insecticide or fungicide, the steps consisting of neutralizing the sulphuric and sulphurous acids present by adding a strong solution of a water-soluble alkali capable of said neutralization, removing the neutralized acid, then agitating the remaining mass with a weaker alkaline water solution, decanting and removing the water solution, then washing the remainder to remove excess alkali, and then adding a small percentage of an insect poison. The insect poison may be nicotine, derris root, or other organic poisons, or inorganic poisons such as copper and arsenic derivatives.

Voorhees (99), assignor to the Standard Oil Company of Indiana, in United States patent 2,011,428, issued August 13, 1935, applied for May 4, 1931, claims "an insecticidal and fungicidal composition comprising a mineral oil, an oil-soluble organic insecticide of vegetable origin which tends to lose its toxicity in the presence of light, and a light-screening oil-soluble organic compound selected from the class consisting of amino anthraquinones."

"Also di-amino and substituted di-amino anthraquinones may be used. The nucleus of the anthraquinone and the organic residue may be substituted with alkyl, aryl, aralkyl, nitro, hydroxyl and other groups. The above class of anthraquinone compounds include phenylated amino anthraquinone, oxphenyl aminanthraquinones, alkyl phenyl amino anthraquinones, diarylamino anthraquinones, 1,4-toluido anthraquinone and aryl diamino anthraquinones."

"The following specific example of a concentrated emulsion suitable for dilution to yield an insecticidal or fungicidal spray is as follows: Gum ghatti, 2.4 percent; cresylic acid, 0.18 percent; water, 35 percent; white oil (30 second Saybolt at 100° F.), 62.4 percent; and 1,4-toluido anthraquinone, 0.02 percent. The concentrated emulsion may be prepared by intimately mixing the ingredients in a colloid mill or by passing the mixture through a centrifugal pump or in any other suitable manner to give a concentrated emulsifiable composition which may readily be diluted to yield an emulsion suitable for spraying purposes."

"The oil may contain the active principles of pyrethrum or other "oil-soluble organic insecticide of vegetable origin" [e.g. derris extract.]

Buc (12), assignor to Standard Oil Development Co., in United States patent 2,013,028, issued September 3, 1935, applied for March 12, 1931, claims an insecticidal composition consisting of a non-volatile viscous petroleum oil containing not less than 0.1 percent of insecticidal material selected from the group consisting of rotenone and rotenoids and not less than 1.5 percent of a non-volatile highly halogenated hydrocarbon. Buc defines rotenoids as the insecticidal principles of certain fish poison plants such as derris and cube which are not exactly identical with rotenone in their chemical constitution but have a similar insecticidal action. A suitable insecticide may be made by dissolving 1 gram rotenone, 20 grams halogenated naphthalene of specific gravity 1.25 in 1 liter kerosene. Other examples are: (1) 2 grams rotenone, 30 grams halogenated naphthalene (halowax oil) of specific gravity 1.25 in 1 liter light lubricating oil; (2) 1.5 grams rotenone, 25 grams of chlorinated gasoline containing 50 percent of chlorine in 1 liter of petroleum oil having a gravity of 27° A.P.I. and a viscosity of 115 Saybolt at 100° F.; (3) an aqueous emulsion containing 0.7 percent of the composition given in Example 1.

Schotte and Gornitz (90), assignors to Schering-Kahlbaum A. G. of Berlin, Germany, in United States patent 2,024,392, issued December 17, 1935, applied for December 12, 1933, in Germany, November 13, 1931, claim an insecticidal preparation consisting of a mixture containing rotenone and veratrin.

They also claim an insecticidal preparation consisting of a mixture containing 2.5 parts radix dorridis ellipticae and 7.5 parts semen sabadillae. The patentees state that by combining one of these drugs containing rotenone with a drug containing veratrin, for instance sabadilla seed or an extract or alkaloids recovered from such products, an insecticidal effect is obtained, which is far greater than the effects exerted by the two drugs if used singly.

"That this is true, is shown by the following comparative tests made by using strewable preparations consisting of intimate ground mixtures of semen sabadillae with talc, radix dorridis ellipticae with talc and with a mixture of semen sabadillae and radix dorridis ellipticae. In each test 30 caterpillars of *Tephrocystia absinthiata* Cl. (Geometridae) placed on blotting paper were dusted with a quantity of one of the mixtures corresponding to .16 milligram per square centimetre, the powder being even distributed.

Preparation	Dead caterpillars	Number of caterpillars not killed
Talc + 2.5% derris root	1	29
Talc + 5% derris root	10	20
Talc + 7.5% derris root	18	12
Talc + 7.5% sabadilla seed	17	13
Talc + 10% sabadilla seed	19	11
Talc + 15% sabadilla seed	26	4
Talc + 2.5% derris root + 7.5% sabadilla seed	30	0
Control test	0	30

A ground mixture of 2.5 parts radix derridis ellipticae, 7.5 parts semen sabadillae and 90 parts talcum proved useful in exterminating grasshoppers (for instance Tachycines asymorus), flea beetles, bugs (Lygus campestris) and many kinds of caterpillars. Instead of the drugs themselves we may also use their effective extracts or the alkaloïds recovered therefrom, for instance by mixing 0.2% rotenone, 0.3% veratrin and 99.5% kaolin."

Hamilton (35), in United States patent 2,030,584, issued February 11, 1936, applied for June 5, 1934, claims an insecticide including water, a suitable oil, and finely powdered natural parts of a plant selected from the group consisting of derris and cube root containing an inherent emulsifier and properties toxic to insects. Reference is made to the use of acetone, alcohol, ether, and ethylene dichlorides as solvents for the active principles of derris and cube. To prepare the paste, 3 or 4 parts of finely powdered derris or cube root are added to 8 parts of water and 8 parts of oil either mineral, vegetable, or animal and the mixture emulsified in a suitable machine. In case the derris is reduced to 1 part it is necessary to add 2/3 to 1 part colloidal clay (wilkeite, bentonite, etc.) to make a physically stable emulsion. A preservative such as 1/2 to 1 percent of carbolic acid may be added; a sodium, potassium, or an ethanalamine soap may be used instead of the clay and an antioxidant such as tannic acid may also be added.

The paste emulsion insecticide produced as aforesaid, makes a quick breaking emulsion when diluted with water for spraying. Insecticide sprays of this invention possess from 15 to 20 percent greater toxicity to plant lice than sprays of the same high dilution made from derris and cube insecticides of the chemical extracted kind.

Hamilton (36), assignor to the Endowment Foundation of New Brunswick, N. J., in United States patent 2,035,546, issued March 31, 1936, applied for May 14, 1932, claims a composition to be used in paste form for killing boring insects including as ingredients a colloidal clay, an insecticide, ethyl alcohol and only sufficient water to form a paste. The insecticide may be an organic insecticidal material such as free nicotine, nicotine sulphate, pyrethrins and rotenone. Rotenone may be used in the form of a solution in acetone, alcohol, carbon tetrachloride, etc.

Buc (13), assignor to the Standard Oil Development Co., in United States patent 2,042,296, issued May 26, 1936, applied for March 12, 1931, claims an insecticidal preparation, comprising a relatively non-volatile petroleum oil, insecticidal material selected from the group consisting of rotenone and rotenoids, and an aryl alkyl ether, the amount of aryl alkyl ether being sufficient to keep the insecticidal material dissolved in the petroleum oil. The following example is given: 0.5 Percent rotenone, 5 percent secondary hexyl cresyl ether, 94.5 percent petroleum oil having a gravity of 27° A. P. I. and a viscosity of 115 Saybolt at 100° F. Buc prepared a stable kerosene solution containing 0.25 percent of rotenone with only 0.75 percent of secondary hexyl phenyl ether as mutual solvent.

Ryan and Moran (83), in U. S. patent 2,043,267, issued June 9, 1936, applied for October 2, 1933, claim a product adapted to serve as concentrate for a germicide, insecticide, or parasiticide, comprising a product which is the result of a chemical reaction between the toxic principle of herbs of the class including pyrethrum flowers and derris root with a chemical compound of the alkylolamine family. For example, derris extract, 1 part, is mixed and heated to about 100° C. with 0.7 part by weight of triethanolamine; then 1 part fatty acid (e. g. oleic) is added. A "disinterested" solvent (one that does not undergo chemical reaction) such as diethylene glycol monoethyl ether is now added in any amount "depending upon the bulk and the ease of solubility desired."

L. E. Smith (94), in U. S. patent 2,049,725 issued August 4, 1936, applied for March 20, 1935, claimed diaryl thioxins, especially phenothioxin, as an insecticide. Upon mosquito larvae, phenothioxin in aqueous solutions in all concentrations up to 1 part in 500,000 was as effective as rotenone.

Wotherspoon (105), assignor to Derris, Inc., in United States patent 2,052,374, issued August 25, 1936, applied for April 28, 1934, claims an aqueous-disseminatable composition of matter consisting of an insecticide obtainable from derris and similar insecticidal-containing plant materials containing rotenone, deguelin, tephrosin, and toxicareol and a water-soluble phenol containing at least two hydroxyl groups.

"I have found that if rotenone, dihydrorotenone, finely ground derris or cube, solid extracts of derris or cube, the hydrogenated derivatives of same, either alone or in mixtures, are melted or intimately mixed with a solid water-soluble phenol containing two or more hydroxyl groups, the resulting product after cooling and grinding is soluble, or miscible, with water in any proportions.

"The products thus produced may be mixed, if desired, with any powdered materials which act as wetting agents, such as soap, alkali salts of sulphonated hydrocarbons or alcohols, sulphonated oils, et cetera.

"Fillers such as starch, glue, gums, dextrans, flour, clays, talc, bentonite, chalk, diatomaceous earth, et cetera, may also be incorporated therein if desired.

"The maximum results are obtained with these insecticides when treated in this manner.

"The phenols referred to above may be one or more of the dihydric phenols such as catechol, resorcinol and quinol and their homologues, and the trihydric phenols such as pyrogallol, phloroglucinol and hydroxyquinol and their homologues."

Examples are: (1) 12.5 parts by weight of rotenone are mixed with 87.5 parts by weight of resorcinol in a vessel equipped with a heating arrangement. Heat is applied and when melted the contents are withdrawn and allowed to cool. The product is then ground in a suitable grinding mill. This material is miscible with water in all proportions and when so mixed is highly toxic to most insects. (2) Twelve and 1/2 parts by weight of dry hydrogenated derris extract are melted with 43.75 parts by weight of resorcinol. The cooled product is ground to a fine powder and mixed with 50 parts by weight of powdered soap. The resulting product when mixed with water is quickly dispersed and yields a most satisfactory horticultural spray. (3) Ten parts by weight of dihydro-rotenone are melted with 90 parts by weight of pyrogallol. The resulting product when ground and dissolved in water yields a valuable insecticide that may be used for mothproofing woolen materials.

Organic solvents mentioned as suitable for dissolving the active principles of Derris, Lonchocarpus, Spatholobus, etc., are ethyl acetate, benzol, acetone, chloroform, ethylene dichloride, and safrol.

Ward (100), in United States patent 2,056,438, issued October 6, 1936, applied for September 4, 1935, in Great Britain, March 30, 1935, claims the process for extracting toxins for the manufacture of insecticides from barbasco root, comprising the steps of treating the root, in a broken up state, with common alcohol in the proportion of 100 grammes of the root to 400 cc. of common alcohol to which is added 1-1/2 percent by volume of chemically pure sulphuric acid, allowing the mixture to digest under the action of the internal heat generated, the common alcohol dissolving out the rotenone, tephrosin, deguelin, and toxicarol, raising the temperature of the mixture by heat derived from an external source, filtering the mixture, neutralizing the filtrate by adding sodium carbonate thereto, refiltering, this final filtrate containing not only the rotenone but also the deguelin, tephrosin, and toxicarol, and mixing the final filtrate with soft soap. Reference is made to the process of obtaining rotenone by extracting the roots with ether and recrystallizing from carbon tetrachloride. This invention relates to the extraction of toxins, for the manufacture of insecticides, from derris, barbasco and timbo roots, such, for example, as Lonchocarpus nicou, Derris elliptica, Derris chinensis, Derris malaccensis and other roots containing rotenone.

Wotherspoon (106), assignor to Derris, Inc., in United States patent 2,058,200, issued October 20, 1936, applied for September 12, 1932, claims a composition of matter consisting of a nonaqueous solution of the insecticidal principles obtainable from one or more members of the group consisting of derris and pyrethrum in at least one member of the group of alkylated

phenolic ethers consisting of safrol, anethol, methyl eugenol, and camphor oil said solvents being in sufficient quantities to maintain the insecticidal principles in solution. Solvents previously used to extract derris and cube include benzene, acetone, acetone-alcohol mixture, petroleum distillate, chloroform, ethylene dichloride, ether, toluene, xylene, etc. Solvents less effective than safrol, etc. include oil of turpentine, pine oil, terpineol, dipentene, phellandrene, terpinolene, limonene, cedrene, and petroleum distillates. The new solvent extracts of derris or cube may also be used in conjunction with water provided a proper colloid is present to form a stable emulsion. These colloids are soap, starch, gum acacia, gum ghatti, gum karaya, gum tragacanth, dextrin, glucose, sucrose, and saponin or extracts of plants rich in the latter.

The following are examples of this invention:

Example 1. 50 parts of a solution of the active principles of derris roots in safrol containing about 10 percent of the derris extractives and 90 percent safrol. 50 parts triethanolamine oleate.

Example 2. 25 parts 5 percent solution of rotenone in camphor oil S. G. 970. 25 parts concentrated solution pyrethrum flowers in camphor oil S. G., 970. 50 parts triethanolamine oleate.

Example 3. 25 parts of a solution of the active principles of derris root containing about 10 percent of the derris extractives and 90 percent safrol. 75 parts of aqueous solution of gum acacia containing 10 percent of the latter. This mixture is passed through a homogenizer or colloid mill to disperse same.

Example 4. 15 parts of a 5 percent solution of rotenone in camphor oil S. G., 970. 10 parts of a concentrated solution of pyrethrum flowers in camphor oils S. G., 970. 25 parts of a concentrated aqueous solution of cocoanut oil soap. 50 parts of an aqueous solution of gum ghatti containing 5 percent of the latter. This mixture is passed through a homogenizer or colloid mill to disperse same.

Example 5. 5 parts of a 2 percent solution of rotenone in safrol are mixed with 95 parts by volume of petroleum distillate.

Example 6. 10 parts of a 1 percent solution of rotenone in camphor oil are mixed with 90 parts by volume of petroleum distillate.

Example 7. 2-1/2 parts of pyrethrum extract (20 to 1) and 5 parts of a 2 percent solution of rotenone in safrol are mixed with 92-1/2 parts by volume of petroleum distillate.

Sankowsky (86), Assignor to Stanco, Inc., in United States patent 2,058,832, issued October 27, 1936, applied for March 17, 1933, claims a method for preparing a clear concentrated liquid insecticide which comprises extracting rotenone and other constituents toxic to insects, from a plant containing such toxic substances, by treating said plant with a mixed solvent, which is capable of selectively dissolving said

toxic substances to the exclusion of resinous materials which, upon dilution of the extract with kerosene, would be precipitated, comprising a petroleum distillate having a maximum boiling point below 625° F. and a sufficient amount of an organic solvent soluble in the petroleum distillate and having solvent power for said toxic substances to dissolve at least 2-1/2 percent of said toxic substances.

Derris root, cube root, or other plants containing rotenone are extracted by a counter-flow method, or by percolation, etc., with about 20 to 30 percent solution of ethylene dichloride in a petroleum distillate. A concentrated extract is obtained containing about 2.5 percent of rotenone and about 5 percent of rotenoids. This concentrated extract, after being clarified if turbid by the addition of a small amount of ethylene dichloride or other similar solvent (e. g. p-dichlorobenzene is mentioned) may be used for the preparation of insecticidal solutions by diluting with a petroleum distillate to the strength desired without the formation of a residue, or it may be first mixed with a concentrated solution of other insecticides and then diluted to the desired strength. The petroleum distillate preferably used is one having a maximum boiling point below 625° F. The term rotenoids is applied to the insecticidal constituents of Derris, Lonchocarpus, and Tephrosia other than rotenone. Other solvents that may be used mixed with a petroleum distillate to extract rotenone and rotenoids from derris root, cube root, or other plants containing rotenone are as follows: acetone, benzol, chlorobenzene, chloroform, dichloroethyl ether, ethylene chlorohydrin, n-propyl formate, toluene, trichloroethylene, and xylene.

Mikeska (74), assignor to the Standard Oil Development Co., in United States patent 2,066,184, issued December 29, 1936, applied for October 29, 1932, claims an improved insecticide, comprising a hydrogenated extract obtained by extracting petroleum oil with a solvent having a preferential solvent action for aromatic and unsaturated hydrocarbons of a petroleum oil, separating the solvent from the extract, hydrogenating the separated extract and dissolving in the hydrogenated extract the active principles of a fish-poisoning plant. For example, kerosene having a solvent power for rotenone and/or rotenoids of from 0.25 to 0.05 percent was extracted with sulphur dioxide. The extract was separated from the solvent by means of distillation and the solvent power for rotenon was found to be increased to 2.3 percent. The extract was then hydrogenated and marked improvements both in the odor and the solvent power were found. The solvent power of the hydrogenated extract for rotenone and/or rotenoids was increased to 7.2 percent. The hydrogenated extract is suitable for use as a solvent in extracting the toxic ingredients from the fish-poisoning plants, by percolation or by maceration of the plant in the presence of the solvent and separating the solvent containing the toxic ingredients from the residual solids.

The hydrogenated extract with the toxic ingredients in solution may be diluted and used as a spray in killing insects, such as flies, mosquitoes, etc. A spray of the emulsion type may be prepared for dipping and spraying animals to control external parasites, such as lice, ticks, mange mites, cattle grub, etc. The emulsion for cattle dips or

sprays is prepared by taking hydrogenated oil extract solution containing 5 to 10 percent of rotenone and/or rotenoids and mixing with water, using a suitable emulsifier such as oil soluble sulphonated soap, fish oil soap, saponin, etc. Other insecticides may be added to the emulsion, such as lead arsenate, nicotine, pyrethrum, etc.

Another use for the spray solution of the emulsion type is for spraying plants such as trees, shrubs, etc. to control parasites such as aphids, leafhoppers, thrips, greenhouse white flies, tent caterpillars, red spiders, squash bugs, roaches, Mexican bean beetles, Japanese beetles, potato beetles, etc.

Rotenoids are defined as the non-crystalline extract of fish-poisoning plants consisting of a mixture of deguelin, tephrosin, toxicarol, etc.

Kilgore (53), in U. S. patent 2,070,603, issued February 16, 1937, applied for January 3, 1936, claims certain esters as insectifuges or insecticides. An example is mesityl oxide oxalate methyl ester. Botanical extractives (e. g. derris extract) themselves toxic to a greater or less degree to insects may be combined with one or more of the compounds described in this patent. In this way the insecticidal and insectifugal values of such extractives are enhanced, and stabilized.

Sessions (91), in U. S. patent 2,083,098, issued June 8, 1937, applied for April 4, 1934, [assignor to the California Spray-Chemical Corp.] claims a parasitocidal composition comprising a neutral copper-calcium silicate containing complex, and a botanical insecticide selected from the group consisting of rotenone, pyrethrum, and nicotine.

Hunn (38), assignor to Stanco, Inc., in United States patent 2,087,599, issued July 20, 1937, applied for September 8, 1932, claims an insecticide including water, a suitable oil and finely powdered natural parts of a plant selected from the group consisting of derris and cube root containing an inherent emulsifier and properties toxic to insects.

The following specific example is given of one way of carrying out the invention. Spent pyrethrum flowers containing approximately 10 percent oil residue extract from a previous extraction of the fresh flowers with oil are pulverized and mixed with water in the proportion of 1 part of flowers to 3 parts of water and about 2 percent of cresylic acid (based on the whole mixture) is added as a preservative. This mixture is, after proper agitation, suitable as a stock emulsion which will be ready for use with mere dilution with water. Ground derris or cube root can be satisfactorily used in a composition similar to the above instead of the spent pyrethrum.

Simanton (93), assignor to the Gulf Research and Development Co., in United States patent 2,089,766, issued August 10, 1937, applied for March 7, 1936, claims:

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"An improved insecticide comprising in stable admixture a petroleum fraction, a substance chosen from the class consisting of ethylene glycol monoethyl ether acetate and diethylene glycol monoethyl ether acetate, pyrethrins, and at least one of the active toxic ingredients of derris and cube."

Chloroform and acetone are mentioned as solvents for rotenone. Among the solvents that have been employed as blending agents in combination with petroleum naphthas are: ethylene dichloride, carbon tetrachloride, acetone, camphor sassafras oil, benzol, and chloroform.

Diethylene glycol monoethyl ether acetate is miscible with a petroleum oil having a viscosity of 100 sec. S. U. V. at 100° F. in concentrations up to 5 percent. With a petroleum oil having a viscosity of 200 sec. S. U. V. at 100° F. the same solvent is miscible in concentrations up to 2 percent. Ethylene glycol monoethyl ether acetate is completely miscible with both of the above oils. Either ethylene glycol monoethyl ether acetate or diethylene glycol monoethyl ether acetate, if miscible with a petroleum fraction in proportion of 1 percent or over, will hold in stable solution toxic amounts of derris extractives. Large proportions of either solvent may be easily incorporated by admixture of light naphthas.

"I find that diethylene glycol monoethyl ether acetate is slightly superior to ethylene glycol monoethyl ether acetate as a solvent for the various extracts of derris and cube, but ethylene glycol monoethyl ether acetate is miscible with the heavier petroleum fractions in larger proportions than is diethylene glycol monoethyl ether acetate. Both compounds are useful."

"Either of the new solvents of my invention is capable of holding 0.05 percent rotenone or other extracts of derris and cube in a petroleum base insecticide when as little as 1 percent is blended with the petroleum oil. Higher percentages will hold greater quantities of derris and cube extracts in solution."

"To be effective as an insecticide, rotenone or other of the derris or cube extracts mentioned should be present in the finished product in concentrations ranging from .01 to 0.1 per cent by weight."

Examples of this invention are:

"In one actual embodiment of the invention I prepared such a solution by first forming a 5 per cent solution of crystallin rotenone in diethylene glycol monoethyl ether acetate and then adding one part of this solution to 100 parts of a petroleum naphtha extract of pyrethrum flowers prepared by extracting 1.2 pounds pyrethrum flowers with a deodorized petroleum naphtha. The resulting product was a stable, non-volatile, substantially odorless solution of excellent insecticidal properties. When tested according to the Peet-Grady method the solution repeatedly gave a knock down value of 98 and a kill of 85.

"In another embodiment of the invention I first prepared a crude extract of dry derris roots by extracting the roots with acetone and

evaporating off the solvent to yield a resinous mass. I made a 5 percent solution of this resinous mass in diethylene glycol monoethyl ether acetate and prepared an insecticidal solution by adding two parts of this 5 per cent solution to 100 parts of a naphtha-pyrethrum extract prepared as before. The resulting insecticidal solution when tested by the Peet-Grady method gave a knock down of 98 and a kill of 88.

"My invention is also applicable to insecticides other than liquid solutions. For instance, the invention is useful when incorporating rotenone or other extracts of derris and cube in insecticidal salves. In one such embodiment of my invention, I first blended 5 parts of a concentrated naphtha solution of pyrethrins with one part of ethylene glycol monoethyl ether acetate containing dissolved therein 5 percent of total extractives prepared by extracting derris with acetone and evaporating off the solvent. I then incorporated this blend with 100 parts of petrolatum by melting the petrolatum, permitting it to cool, and adding the blend while stirring just before the petrolatum became solid. The resulting mixture was a stable odorless insecticide of salve-like homogeneous consistency. This salve is useful for application to the ears of cattle to kill ear ticks."

Coe (16), in United States patent 2,090,109, issued August 17, 1937, applied for November 15, 1933, claims

"A composition of matter comprising a suspension of a powdered insecticide of plant origin subject to deterioration by ordinary light, in a colored globule forming liquid of adhesive tendency and non-repellent to insects, the color of the globule forming liquid having a spectral transmission of above 4900 Angström units of the visible spectrum."

For example a liquid gelatin is formed by dissolving the gelatin in water. The resulting liquid gelatin is then colored with a water soluble chlorophyll green dye which may be made of thiocarmine blue and tartrazine yellow. Powdered rotenone is then added to the chlorophyll green liquid gelatin. The amount of rotenone to be used is governed by the desired toxic strength of the insecticide, and sufficient water is added to bring the suspension to the desired consistency for spraying purposes.

The color screen may be formed of an orange yellow dye, or the colors above 4900 Angström units of the visible spectrum, although chlorophyll green and orange yellow are preferable. A black pigment or opaque substance may be used in forming the color screen by the use of which all light is excluded from the particles of the substance being protected.

Fawcett (25), assignor to Imperial Chemical Industries Ltd., in United States patent 2,096,678, issued October 19, 1937, applied for October 25, 1935, in Great Britain October 29, 1934, makes essentially the same claims as in British patent 446,576.

Donlan (20), assignor to Stanco, Inc., in United States patent 2,096,885, issued October 26, 1937, applied for October 21, 1933, claims an insecticidal composition comprising as an active ingredient a substantial quantity of mineral oil of 300° to 600° F. boiling range consisting predominantly of paraffinic hydrocarbons segregated from the normally accompanying naphthenic hydrocarbons and added active insecticidal principles of vegetable origin, e.g., pyrethrum, cube, tuba, and haiari.

Jones (52), in United States patent 2,103,195, issued December 21, 1937, applied for July 3, 1937, dedicated to the free use of the people of the United States of America, claims a new chemical combination or rotenone and dichloroacetic acid having the empirical formula $C_{23}H_{22}O_6 \cdot C_2H_2O_2Cl_2$. He also claims a process for making a chemical compound of rotenone and dichloroacetic acid, which comprises essentially dissolving an extract of plant material (e.g. Derris, Lonchocarpus, Tephrosia) containing rotenone in dichloroacetic acid, adding water and separating the resulting crystalline product. Reference is made to the addition compounds of rotenone with acetic acid, propionic acid and alpha-chloropropionic acid in each of which the molecular ratio is two mols of rotenone to one mol of acid.

O'Kane (80), in United States patent 2,104,757, issued January 11, 1938, applied for May 31, 1934, claims as an insecticidal agent, a substantially nonaqueous combination of an oil selected from the class consisting of vegetable and animal oils, a contact insecticide selectively soluble in said oil, and an emulsifying agent soluble in said contact agent in proportions to give an unstable dispersion in water.

Among the insect poisons, a wide variety of materials may be included, and particularly exemplifying the nerve poisons or contact insecticides, there may be mentioned the extracts of pyrethrum flowers, and other oil-soluble extracts, such as those of derris root, cube root, or oil-soluble nerve poisons, such as the active principle of strychnia, as well as synthetic chemicals including various amine derivatives.

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29. GOLDSCHMIDT, S., and MARTIN, K.

1936. Insecticide. Canadian Patent 357,959; issued May 19, 1936; applied for December 17, 1934; in Germany December 27, 1933.

30. GRAAFLAND, A. J. N., and NELLENSTEYN, F. J.

1931. Antifouling composition. British Patent 362,526; issued December 10, 1931; applied for September 10, 1930; in Holland, September 12, 1929.

31. GRANT, D. H.

1929. Insecticide. Canadian Patent 333,642; issued June 27, 1933; applied for October 25, 1929.

32. -----

1933. Insecticide. U. S. Patent 1,934,057; issued November 7, 1933; applied for October 31, 1928; assigned to Standard Oil Development Co.

33. -----

1933. Insecticide and process of making the same. U. S. Patent 1,940,646; issued December 19, 1933; applied for October 19, 1927; assigned to Standard Oil Development Co.

34. HALLER, H. L. J. and SCHAFER, P. S.

1934. Process of preparing dihydrorotenone. U. S. Patent 1,945,312; issued January 30, 1934; applied for April 10, 1933.

35. HAMILTON, C. C.

1936. Insecticide. U. S. Patent 2,030,584; issued February 11, 1936; applied for June 5, 1934.

36. -----

1936. Insecticide composition. U. S. Patent 2,035,546; issued March 31, 1936; applied for May 14, 1932.

37. HOFFMANN, F.-LA ROCHE AND CO.

1926. Schadlingsbekämpfungsmittel. Swiss Patent 124,290; issued January 16, 1928; applied for October 5, 1926.

38. HUNN, E. B.

1937. Horticultural oil spray. U. S. Patent 2,087,599; issued July 20, 1937; applied for September 8, 1932.

39. I. G. FARBENINDUSTRIE A. G.

1932. Improved preparations for killing flies. British Patent 377,069; issued July 21, 1932; applied for November 27, 1931; in Germany, November 27, 1930.

40. -----

1932. Fliegenvertilgungsmittel. German Patent 555,117; issued July 18, 1932; applied for November 28, 1930

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1932. Improved process for killing or eliminating flies and preparations therefor. British Patent 378,300; issued August 11, 1932; applied for December 12, 1931; in Germany, December 13, 1930.

42. IIBA, T., and NAKATUKA, S.

1931. Insecticide. Japanese Patent 91,478; issued May 20, 1931; [Abstract in Chem. Abs. 26;1703. 1932.]

43. IMAI, Y.

1931. Insecticide. Japanese Patent 91,645; issued June 3, 1931; [Abstract in Chem. Abs. 26;1703. 1932.]

44. IMPERIAL CHEMICAL INDUSTRIES, LTD.

1936. Procédé de fabrication de la rotenone et des substances associées en partant des produits naturels qui en contiennent. French Patent 797,052; issued April 20, 1936; applied for October 29, 1935; in England, October 29, 1934.

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1936. Improvements in or relating to the protection of sheep and cattle from infestation by insects. Australian Patent 20,894/35; issued February 14, 1935; applied for January 11, 1935.

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1937. Rotenone production. Canad. patent 369,499; issued October 26, 1937; applied for October 28, 1935. Assignee of E. W. Fawcett.

47. ISIHARA, T.

1931. Insecticide. Japanese Patent 91,875; issued June 20, 1931; [Abstract in Chem. Abs. 26;1703. 1932.]

48. JAMES, J. H.

1935. Manufacture of insecticides and fungicides. U. S. Patent 2,006,456; issued July 2, 1935; applied for March 12, 1930.

49. JONES, H. A.

1933. Insecticide and process for making the same. U. S. Patent 1,928,256; issued September 26, 1933; applied for August 23, 1930.

50. -----

1933. Process for making an insecticide. U. S. Patent 1,928,968; issued October 3, 1933; applied for August 23, 1930.

51. -----

1934. Process of extracting rotenone from plant material. U. S. Patent 1,942,104; issued January 2, 1934; applied for February 20, 1933.

52. -----

1937. Insecticide. U. S. Patent 2,103,195; issued December 21, 1937; applied for July 3, 1937.

53. KILGORE, L. B.

1937. Insectifuge and insecticide. U. S. Patent 2,070,603; issued February 16, 1937; applied for January 3, 1936.

54. LEMMENS, G. J., and FRYER, P. J.

1917. Improvements relating to insecticides, sheep dips, animal washes and the like. British Patent 105,525; issued April 19, 1917; applied for December 4, 1916.

55. -----, . . . and FRYER, P. J.

1920. Mittel zur vertilgung von insekten und sonstigen auf tieren oder pflanzen wohnenden parasiten. German Patent 321,317; issued May 29, 1920; applied for December 21, 1916.

56. ----- and FRYER, P. J.

1920. Mittel zur vertilgung von insekten und sonstigen auf tieren oder pflanzen wohnenden parasiten. German Patent 327,583; issued October 11, 1920; applied for December 22, 1916.

57. LINGLER, J.

1931. Method of fumigating with calcium cyanide. U. S. Patent 1,820,394; issued August 25, 1931; applied for March 9, 1929; in Germany, March 12, 1928.

58. McDOUGALL, I., and HOWLES, F.

1923. Improvements in the manufacture of insecticides, sheep dips, vermifuges and the like. British patent 226,250; issued December 23, 1924; applied for June 23, 1923.

59. ----- and HOWLES, F.

1924. Improvements in the manufacture of insecticides, sheep dips, vermifuges and the like. Australian Patent 18,227; issued November 10, 1924; applied for June 16, 1924.

60. ----- and HOWLES, F.

1925. Improvements relating to insecticides. British Patent 233,857; issued May 21, 1925; applied for April 15, 1924.

61. ----- and HOWLES, F.

1925. Insecticide, sheep dip, vermifuge, etc. Canadian Patent 247,627; issued March 17, 1925; applied for May 22, 1924.

62. ----- and HOWLES, F.

1925. Perfectionnements dans la fabrication d'insecticides, de bains pour moutons, de vermifuges et de preparations similaires. French Patent 589,969; issued June 9, 1925; applied for May 14, 1924.

63. McDOUGALL, I. S., McDOUGALL, I., and McDOUGALL, S.

1912. Improvements relating to insecticides, sheep dip, vermifuges, and the like. British Patent 3,204 of 1911; issued May 8, 1912; applied for February 8, 1911.

64. ----- McDOUGALL, I., and McDOUGALL, S.

1912. Improvements relating to insecticides, sheep dip, vermifuges and the like. British Patent 8,322; issued December 19, 1912; applied for April 6, 1912.

65. ----- McDOUGALL, I., and McDOUGALL, S.

1912. Improvements relating to insecticides, sheep dips, vermifuges and the like. British Patent 10,215; issued May 28, 1912; applied for April 27, 1911.

66. ----- McDOUGALL, I., and McDOUGALL, S.

1913. Insecticide, sheep-dip, vermifuge, etc. U. S. Patent 1,065,156; issued June 17, 1913; applied for September 8, 1911

67. McDOUGALL AND YALDING (LTD.)

1925. Insecticide, sheep dips and the like. Canadian Patent 253,319; issued September 1, 1925; applied for November 26, 1924.

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1925. Perfectionnements aux insecticides, aux bains pour moutons et aux substances similaires. French Patent 589,506; issued May 30, 1925; applied for November 22, 1924.

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1925. Improvements in insecticides, sheep dips, and the like. British Patent 229,773; issued March 2, 1925; applied for December 1, 1923.

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1926. Improvements in insecticides, sheep dips and the like. British Patent 246,252; issued January 28, 1926; applied for November 12, 1924.

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72. MCGILL, W. J.

1932. Mothproofing. U. S. Patent 1,854,948; issued April 19, 1932; applied for November 7, 1927.

73. -----

1934. Mothproofing composition. Canadian Patent 338,090; issued January 23, 1934; applied for April 1, 1932; assigned to Standard Oil Co.

74. MIKESKA, L. A.

1936. Insecticide and insect repellent and method of making the same. U. S. Patent 2,066,184; issued December 29, 1936; applied for October 29, 1932.

75. MILLS, L. E. and FAYERWEATHER, B. L.

1934. Tertiary alkyl substituted orthodihydroxybenzenes. U. S. Patent 1,942,827; issued January 9, 1934; applied for March 27, 1933.

76. NETTAI SANGYO KABUSHIKI KAISHA

1926. A new or improved process for obtaining insecticidal or other toxic substances from plants of the derris species. British Patent 247,140; issued August 5, 1926; applied for June 25, 1925; in Japan, February 4, 1925.

77. -----

1930. Werkwijze voor het bereiden van een-insecticide, dat een werkzaam bestanddeel van derrissoorten bevat, verkregen door extractie met organische oplosmiddelen. Dutch Patent 21,841; issued May 15, 1930; applied for February 10, 1926.

78. OHORI, T., and NAKAZAWA, K.

1930. Process for producing a liquid insecticide containing effective ingredient of derris species. U. S. Patent 1,745,907; issued February 4, 1930; applied for February 10, 1928; in Japan, February 18, 1927; assigned to Zaiden Hojin Rikagaku Kenkujo, Tokyo, Japan.

79. O'KANE, W. C.

1930. Fungicide and insecticide. U. S. Patent 1,786,125; issued December 23, 1930; applied for May 18, 1927.

80.

1938. Insecticide spray. U. S. Patent 2,104,757; issued January 11, 1938; applied for May 31, 1934.

81. PFAFF, K. and DUNKEL, M.

1934. Insecticidal agent. Canadian Patent 345,383; issued October 16, 1934; applied for December 12, 1931; in Germany, December 13, 1930.

82. RIEDEL-E. DE HAEN, J. D., A.-G.

1933. Procédé d'aneantissement de la vermine, en particulier d'insectes. French Patent 778,844; issued March 25, 1935; applied for September 24, 1934; in Germany, September 30, and November 8, 1933.

83. RYAN, V. H., and MORAN, J. A.

1936. Preparation of insecticides. U. S. Patent 2,043,267; issued June 9, 1936; applied for October 2, 1933.

84. SANDERS, G. E.

1931. Process of forming colloidal suspensions containing copper and product thereof. U. S. Patent 1,803,870; issued May 5, 1931; applied for October 3, 1927.

85. -----

1932. Method of making insecticides floatable. U. S. Patent 1,847,540; issued March 1, 1932; applied for March 11, 1930.

86. SANKOWSKY, N. A.

1936. Insecticides containing derris extracts and methods of preparing the same. U. S. Patent 2,058,832; issued October 27, 1936; applied for March 17, 1933.

87. ----- and FULTON, S. C.

1934. Insecticide. Canadian Patent 340,315; issued March 20, 1934; applied for December 24, 1931; in the United States January 5, 1931.

88. SCHOTTE, H. and GORNITZ, K.

1935. Insecticide. Canadian Patent 348,379; issued February 26, 1935; applied for January 24, 1934.

89. ----- and GORNITZ, K.

1935. Insecticide. Canadian Patent 348,380; issued February 26, 1935; applied for January 24, 1934.

90. ----- and GORNITZ, K.

1935. Insecticidal preparation. U. S. Patent 2,024,392; issued December 17, 1935; applied for December 12, 1933; in Germany, November 13, 1931.

91. SESSIONS, A. C.

1937. Parasiticial composition. U. S. Patent 2,083,098; issued June 8, 1937; applied for April 4, 1934; assigned to California Spray-Chemical Corp.

92. SHELL DEVELOPMENT COMPANY

1937. Insecticide. Canad. Patent 369,390; issued October 19, 1937; applied for December 24, 1936; in United States, January 6, 1936.

93. SIMANTON, W. A.

1937. Insecticide. U. S. Patent, 2,089,766; issued August 10, 1937; applied for March 7, 1936.

94. SMITH, L. E.

1936. Insecticide. U. S. Patent 2,049,725; issued August 4, 1936; applied for March 20, 1935.

95. STANDARD OIL DEVELOPMENT COMPANY

1931. Improved insecticidal, insect-repelling and fungicidal compositions. British Patent 350,897; issued June 15, 1931; applied for February 15, 1930.

96. ----- SANKOWSKY, N. A., and FULTON, S. C.

1933. Improvements in or relating to insecticides. British Patent 394,977; issued July 4, 1933; applied for January 4, 1932; in the United States, January 5, 1931.

97. STEINDORFF, A., PFAFF, K., KRAMER, R., and KERN, H.

1931. Mittel zur bekämpfung von musciden und sonstigen insekten. German Patent 564,018; issued November 12, 1932; applied for July 5, 1931.

98. TAKEI, S.

1929. Process for producing a liquid insecticide containing effective ingredient of derris species. U. S. Patent 1,724,626; issued August 13, 1929; applied for August 31, 1925; in Japan, September 8, 1924.

99. VOORHEES, V.

1935. Light stable insecticide. U. S. Patent 2,011,428; issued August 13, 1935; applied for May 4, 1931; assigned to Standard Oil Co., of Indiana, Chicago, Ill.

100. WARD, H. G.

1936. Extraction of toxins from roots for the manufacture of insecticides. U. S. Patent 2,056,438; issued October 6, 1936; applied for September 4, 1935; in Great Britain March 30, 1935.

101. -----

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102. WIKSTROM, E. G. A.

1935. Insecticide and method of producing the same. British Patent 434,660; issued September 6, 1935; applied for March 19, 1934.

103. -----

1936. Insektdödande medel. Swedish Patent 86,083; issued April 15, 1936; applied for March 16, 1934.

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1936. Insektdödande medel. Swedish Patent 87,625; issued October 13, 1936; applied for March 16, 1934.

105. WOTHERSPOON, R.

1935. Process for producing water-soluble and water miscible insecticides and products produced thereby. U. S. Patent 2,052,374; issued August 25, 1935; applied for April 28, 1934.

106. -----

1936. Insecticidal solution. U. S. Patent 2,058,200; issued October 20, 1936; applied for September 12, 1932.

107. ZAIDAN HOJIN RIKAGAKU KENKYUJO

1926. A process for producing a liquid insecticide containing effective ingredient of derris species. British Patent 239,483; issued May 6, 1926; applied for April 24, 1925; in Japan, September 8, 1924.

108. -----

1928. A process for producing a liquid insecticide containing effective ingredient of derris species. British Patent 285,797; issued November 29, 1928; applied for February 20, 1928; in Japan, February 18, 1927.

109. -----

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110. -----

1929. Powdered insecticide containing active ingrediants of roots of derris species. Australian Patent 15,327/28; issued July 24, 1929; applied for August 28, 1928; in Japan, November 16, 1927.

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Anderson, W. J.; see Dougherty, L. B.
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 sulphonated, U. S. 2,052,374, 1,940,646, 1,583,681
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 vegetable hard, U. S. 1,724,626
 vegetable hydrogenated, Brit. 239,483
 vegetable sulphonated, U. S. 1,940,646
 whale, sperm, Canad. 253,319; French 589,506; U. S. 1,583,681

Paraffin, Brit. 239,483; Dutch 21,841; U. S. 1,724,626
Paraffin wax, sulphonated, Canad. 253,319; French 589,506; U. S. 1,583,681
Peanut oil, French 769,445
Potrolatum, U. S. 2,089,766
Petroleum -
 fractions, sulphonated, U. S. 2,006,456
 sulfonates, U. S. 1,934,057
 sulphuric soap acid, Brit. 350,897
 white oil, Brit. 350,897
Phenols, U. S. 1,940,646, 2,030,584
Phenols, dihydric, U. S. 2,052,374
Phenols, trihydric, U. S. 2,052,374
Phenyl salicylate, Ger. 555,117
Phloroglucinol, U. S. 2,052,374
Phytosterinoxide, Swiss 124,290
Pine resin, Brit. 239,483; Dutch 21,841; U. S. 1,724,626
Potassium oleate, U. S. 1,928,968
Protein, animal, Dutch 21,841
Pyrethrins, U. S. 1,967,024, 2,058,200, 2,089,766
Pyrethrum, U. S. 1,940,899, 2,066,184
Pyrethrum, extract, Brit. 434,660, Canad. 369,390
Pyrogallol, U. S. 2,052,374
Quassia extract, Brit. 434,660; French 769,445
Quinol, U. S. 2,052,374
Resins, Brit. 285,797; 239,483; Dutch 21,841; U. S. 1,724,626
Resorcinol, U. S. 2,052,374
Rosin soap, Brit. 350,897; U. S. 1,934,057, 1,940,646
Sabadilla seeds, Canad. 348,379; U. S. 2,024,392
Saponin, Brit. 350,897; U. S. 1,928,968; 1,934,057, 2,058,200, 2,066,184
Siliceous earth, U. S. 1,940,899
Soaps - Brit. 239,483, 285,797; 434,660; Dutch 21,841; Swedish 86,083; U. S.
 1,621,340, 1,724,626, 1,745,907, 1,934,057, 2,052,374, 2,058,200
Soaps, fatty, U. S. 1,934,057
Soaps, fatty acid, Brit. 350,897
Soaps, fish oil, U. S. 2,066,184
Soaps, oil-soluble sulphonated, U. S. 2,066,184
Soaps, potassium, U. S. 2,030,584
Soaps, petroleum sulphonic acid, Brit. 350,897
Soaps, resin, Brit. 432,626, 434,660
Soaps, sodium, U. S. 2,030,584
Soaps, soft, Brit. 437,171; U. S. 1,065,156, 2,056,438
Soaps, sulphonated vegetable oil, U. S. 1,934,057
Sodium carbonate in mfg. extract, U. S. 2,056,438
Soy bean -
 milk, Dutch 21,841
 oil, Dutch 21,841
Starch, Brit. 239,483; Dutch 21,841, U. S. 1,724,626, 1,928,968, 2,052,374,
 2,058,200
Stearic acid, U. S. 1,847,540
Sucrose, U. S. 2,058,200
Suet, hog, Dutch 21,841

Sulphonated alcohols, U. S. 2,052,374
Sulphonated animal oil soaps, U. S. 1,934,057
Sulphonated fatty body, U. S. 1,583,681
Sulphonated oil soluble, U. S. 1,940,646
Sulphur, U. S. 1,065,156
Talc, U. S. 1,940,899, 2,024,392, 2,052,374
Talcum powder, U. S. 1,621,340
Tallow, beef, Brit. 239,483; Dutch 21,841; U. S. 1,724,626
Tannic acid, U. S. 1,928,968, 2,030,584
Terpineol, U. S. 1,940,646
Triethanolamine, Brit. 434,660
Triethanolamine, oleate, French 769,445; U. S. 1,928,968, 2,043,267, 2,058,200
Turkey red oil, U. S. 1,940,646
Turpentine, Japanese, 91,645
Urethanes - Ger. 564,018
 substituted, Ger. 564,018
Veratrin, Can. 348,379; U. S. 2,024,392
Water softening agent, U. S. 1,065,156, Brit. 3204/1911
Waxes, Brit. 239,483, 285,797; Dutch 21,841; U. S. 1,724,626
Wilkeite, U. S. 2,030,584
Wood wax, Brit. 239,483; Dutch 21,841; U. S. 1,724,626
Wyrol, U. S. 1,934,057

Extractants and Solvents

Acetic acid, U. S. 1,928,968, 2,103,195
 dichloro, U. S. 2,103,195
Acetone, U. S. 1,928,968, 1,934,057, 1,945,312, 2,030,584, 2,035,546,
 2,052,374, 2,058,200, 2,089,766
Acetone plus alcohol, U. S. 2,058,200
Acetone plus ether, Swiss 124,290
Acetyl acetone, U. S. 1,928,968
Alcohol, Brit. 432,626, 437,171; U. S. 1,621,240, 1,854,948, 1,945,312,
 2,030,584, 2,035,546
Alcohol plus ether, Swiss, 124,290
Alcohol plus sulphuric acid, U. S. 2,056,438
Alcohols, Brit. 350,897; U. S. 1,934,057, 1,940,646
Alkyl pyridines, U. S. 1,928,256
Allyl pyridine, U. S. 1,928,256
Anethol, U. S. 2,058,200
Animal Oil, U. S. 2,104,757

Apiezon, Brit. 446,576
Benzene, Brit. 246,252; U. S. 1,583,681; 1,934,057, 1,945,312, 2,058,200
Benzene, p-dichloro, U. S. 2,058,832
Benzene, Brit. 3204/1911, 432,628, 105,525; U. S. 1,065,156
Benzol, Brit. 285,797, 350,897; U. S. 1,621,240, 1,745,907, 1,854,948;
 1,934,057, 2,934,057, 1,940,646, 1,967,024, 2,052,374
Bipyridine, U. S. 1,928,200, 1,928,256
Brown coal oil, Brit. 378,300

Camphor oil, Dutch 21,841; U. S. 1,724,626, 2,058,200
Carbon tetrachloride, Brit. 239,483, 285,797, 432,626; Dutch 21,841;
U. S. 1,724,626, 1,745,907 1,940,646, 1,942,104, 1,967,024, 2,035,546,
2,056,438
Cedrene, U. S. 2,058,200

Chloroform, Brit. 239,483; Dutch 21,841; U. S. 1,724,626, 2,052,374, 2,058,200
2,089,766
Citron oil, Dutch 21,841

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coal tar oil, Brit. 378,300
Cottonseed oil, Brit. 446,576
Cresol, Brit. 280,256, 350,897; U. S. 1,934,057
Cresol, homologues, U. S. 1,934,057
Cyclohexanol, Brit. 350,897; U. S. 1,934,057
Cyclohexanol homologues, U. S. 1,934,057
Cymene, Brit. 434,660; Swed. 86,083
Decalin, U. S. 1,934,057
Diacetone alcohol, U. S. 1,928,968
Dipentene, U. S. 2,058,200

Esters, Brit. 350,897; U. S. 1,934,057
Ethane, trichloro, Brit. 285,797; U. S. 1,745,907
Ethane, trichloro, plus methyl alcohol, Japan. 91,478
Ether, Austral. 15,327/28; Brit. 239,483, 285,797; Dutch 21,841; Swiss 124,290;
U. S. 1,724,626, 1,745,907, 1,854,948, 1,945,312, 2,030,584, 2,056,438,
2,058,200
Ether arylalkyl, U. S. 2,042,296
Ether diethylene glycol, monoethyl, U. S. 2,039,766, 2,043,267
Ether, sec-hoxylcresyl ether, U. S. 2,042,296
Ether, sec-hexyphenyl ether, U. S. 2,042,296
Ethers, Brit. 350,897; U. S. 1,934,057
Ethyl, Dutch 21,841; U. S. 1,724,626, 1,928,256
Ethyl acetate, U. S. 1,945,312, 2,052,374
Ethyl alcohol, Dutch 21,841; U. S. 1,724,626, 1,928,968, 1,940,646
Ethyl alcohol, absolute, Brit. 239,483
Ethyl formate, U. S. 1,928,968
Ethylene chlorohydrin, U. S. 1,514,377, 1,928,968
Ethylene dichloride, U. S. 2,030,584, 2,052,374, 2,058,200
Ethylene, dichloro, Brit. 423,625, 432,626
Ethylene, trichloro, Brit. 432,626
Gasoline, chlorinated, U. S. 2,013,023
Glycol, diethylene, monoethyl ether, U. S. 2,043,267, 2,089,766
Halohydrin, U. S. 1,514,377
Hallowax oil, Brit. 350,897; U. S. 1,934,057
Hydrocarbon, aliphatic, Ger. 643,804
Hydrocarbon, aromatic, Brit. 350,897; U. S. 1,583,057
Hydrocarbon oil, Brit. 280,256
Hydrocarbon white oil, U. S. 1,786,125
Hydrocarbon white oil, chlorinated, Canada. 338,896; U. S. 1,934,057

Hydrocarbons -

chlorinated, Canad. 338,896; U. S. 1,934,057

Paraffinic, U. S. 2,096,885

Unsaturated, Brit. 394,977; Canad. 340,315

Hydrogenated shale oil, Brit. 378,300

Hydrogenated solvent naphtha, Brit. 378,300

Isopropyl alcohol, Brit. 350,897; U. S. 1,934,057, 1,940,646.

Kerosene, Brit. 446,576; U. S. 1,940,646, 1,967,024, 2,013,038, 2,042,296

Kerosene plus chloroform, U. S. 1,967,024

Kerosene plus oil soluble sulfonate, U. S. 1,940,646

Kerosene plus oil soluble sulfonate plus ethyl methyl ketone, U. S. 1,940,646

Kerosene plus sec-butyl alcohol, U. S. 1,940,646

Ketone, dicthyl, U. S. 1,928,968

Ketones, Brit. 350,897; U. S. 1,934,057

Lemon oil, U. S. 1,724,626

Lignite tar oil, Brit. 378,300

Limonene, U. S. 2,058,200

Linseed stand oil, Brit. 446,576

Lubricants, oils, Brit. 280,256

Lutidine, (dimethyl pyridine) U. S. 1,928,256

Mesityl oxide oxalate methyl ester, U. S. 2,070,603

Methanol (methyl-alcohol), Brit. 239,483; Dutch 21,841; U. S. 1,724,626

Methyl eugenol, U. S. 2,058,200

Methyl ethyl acetone, U. S. 1,928,968

Methyl ethyl ketone, U. S. 1,928,968

Mineral oil, U. S. 2,011,428

Mineral oil b.p. 300-600° F., U. S. 2,096,885

Mineral oil distillate, Canad. 369,390

Mineral oil, heavy, Brit. 446,576

Mineral oil, hydrogenated, Canad. 345,383

Mineral oil, volatile, Brit. 446,576

Mineral seal oil plus oil soluble sulphonate plus potash rosin soap plus
ethyl methyl ketone, plus hexahydrophenol, plus cresol, U. S. 1,940,646

Naphtha, U. S. 1,940,646

Naphthalenes, chlorinated, Brit. 350,897; U. S. 1,934,057

Naphthalenes, hydrogenated, Brit. 350,897; U. S. 1,934,057

Oils -

pine, U. S. 2,058,200

pine, steam distilled, Brit. 350,897; U. S. 1,934,057

Olive oil, Brit. 446,576

Petroleum - Ger. 643,804

Petroleum distillate, U. S. 2,058,200

Petroleum oil, Brit. 377,069; Ger. 555,117

Petroleum distillate plus -

acetone, U. S. 2,058,832

benzol, U. S. 2,058,832

chlorobenzene, U. S. 2,058,832

chloroform, U. S. 2,058,832

dichloroethyl ether, U. S. 2,058,832

ethylene dichloride, U. S. 2,058,832
ethylene chlorohydrin, U. S. 2,058,832
n-propyl formate, U. S. 2,058,832
toluene, U. S. 2,058,832
trichloroethylene, U. S. 2,058,832
xylene, U. S. 2,058,832
Petroleum ether, Brit. 239,483; Dutch 21,841; U. S. 1,724,626
Petroleum naphtha, Canad. 338,896, 338,897; U. S. 1,854,948
Petroleum naphtha plus --
acetone, U. S. 2,089,766
benzol, U. S. 2,089,766
camphor sassafras oil, U. S. 2,089,766
carbon tetrachloride, U. S. 2,089,766
chloroform, U. S. 2,089,766
diethylene glycol monoethyl ether acetate, U. S. 2,089,766
ethylene dichloride, U. S. 2,089,766
ethylene glycol monoethyl ether acetate, U. S. 2,089,766
Petroleum oil --
hydrogenated, U. S. 2,066,184
non-volatile, U. S. 2,042,296
non-volatile viscous, U. S. 2,013,028
white, Brit. 350,897
Petroleum white oil, Brit. 350,897; U. S. 1,934,057
Petroleum white oil plus chloronaphthalene plus K-rosin soap, plus oil soluble
sulphonate, plus sec-amyl alcohol, plus steam distilled pine oil,
U. S. 1,940,646
Petroleum white oil plus oil soluble sulphonate plus crude dichlorobenzene,
U. S. 1,940,646
Phellandrene, U. S. 2,058,200
Phenol, Brit. 350,897; U. S. 1,934,057
Phenol ether, alkylated, U. S. 2,058,200
Phenols, Brit. 280,256
Phenyl ethyl ketone, U. S. 1,928,968
m-phenyl tolyl ketone, U. S. 1,928,968
o-phenyl tolyl ketone, U. S. 1,928,968
alpha-picoline, U. S. 1,928,256
beta-picoline, U. S. 1,928,256
gamma-picoline, U. S. 1,928,256
Propionic acid, U. S. 2,103,195
Propionic acid, alpha chloro, U. S. 2,103,195
Propylene chlorohydrin, U. S. 1,514,377
Pyridine, U. S. 1,928,256, 1,928,968
Pyridine, alkyl, U. S. 1,928,256
Pyridine allyl, U. S. 1,928,236
Pyridine, ethyl, U. S. 1,928,256
Pyridine base, U. S. 1,928,256
Pyridine, dimethyl, U. S. 1,928,256
Pyridine, trimethyl, U. S. 1,928,256
Safrol, U. S. 2,052,374, 2,058,200
Shale oil, hydrogenated, Brit. 378,300
Sodium bisulphite solution, Ger. 630,483

Terpineol, Brit. 350,897; U. S. 1,934,057, 2,058,200
Terpinolene, U. S. 2,058,200
Tetralin, U. S. 1,934,057
Toluene, U. S. 2,058,200
Turpentine, oil, U. S. 2,058,200
Vegetable oil, U. S. 2,104,757

white oil, Brit. 350,897; Canad. 333,642; U. S. 2,011,428
Xylene, U. S. 2,058,200

Reagents Used in Preparing Derris Extract

Aluminum salt for coagulating derris latex	Brit. 226,250
Aluminum sulphate for coagulating derris latex	Brit. 226,250
Iron salt for coagulating derris latex	Brit. 226,250
Lead salt for coagulating derris latex	Brit. 226,250
Lime, milk of, for coagulating derris latex	Brit. 214,822
Sulphuric acid in alcohol for extracting derris	Brit. 437,171
Salt in preparing derris extract	Brit. 214,822
Sodium carbonate in preparing derris extract	Brit. 214,822, 437,171
Sodium hydroxide in preparing derris extract	Brit. 226,250
Tartaric acid in preparing derris extract	Brit. 214,822

Stabilizers

Anthracene, Brit. 350,897
Anthraquinones, alkyl, U. S. 2,011,428
Anthraquinones, amino, U. S. 2,011,428
Anthraquinones, amino, alkylphenyl, U. S. 2,011,428
Anthraquinones, amino, diaryl, U. S. 2,011,428
Anthraquinones, amino, oxyphenyl, U. S. 2,011,428
Anthraquinones, amino, phenylated, U. S. 2,011,428
Anthraquinones, aralkyl, U. S. 2,011,428
Anthraquinones, aryl, U. S. 2,011,428
Anthraquinones, aryl diamino, U. S. 2,011,428
Anthraquinones, diamino, U. S. 2,011,428
Anthraquinones, hydroxy, U. S. 2,011,428
Anthraquinones, nitro, U. S. 2,011,428
Anthraquinones, 4,4-toluido, U. S. 2,011,428
Benzene, 1,2-dihydroxy-4-tert. alkyl, U. S. 1,942,827
Benzene, 1,2-dihydroxy-4-tert. amyl, U. S. 1,942,827
Benzene, 1,2-dihydroxy-4-tert butyl, U. S. 1,942,827
Black pigment, U. S. 2,090,109
Camphor, Brit. 350,897
Chlorophyll green dye, U. S. 2,090,109
Diphenyl, Brit. 350,897
Furoin, Brit. 350,897
alpha-Naphthol, Brit. 350,897
Nitrocresol, Brit. 350,897
Orange yellow dye, U. S. 2,090,109

Phenanthrene, Brit. 350,897
Phenylhydrazine, Brit. 350,997
Toluidine, Brit. 350,897

A List of All Patents Arranged According to Filing Dates

<u>Filing Date</u>	<u>Patent</u>
February 8, 1911	Brit. 3,204/1911
April 27, 1911	Brit. 10,215/1911
September 8, 1911	U. S. 1,065,156
April 6, 1912	Brit. 8,322/1912
December 4, 1916	Brit. 105,525
December 21, 1916	Ger. 321,317
December 22, 1916	Ger. 327,583
March 31, 1920	U. S. 1,514,377
April 16, 1923	Brit. 214,822
April 16, 1923	U. S. 1,621,240
June 23, 1923	Brit. 226,250
December 1, 1923	Brit. 229,773
April 15, 1924	Brit. 233,857
May 14, 1924	French 589,969
May 22, 1924	Canad. 247,627
June 16, 1924	Austral. 18,227/24
July 22, 1924	U. S. 1,522,041
November 12, 1924	Brit. 246,252
November 22, 1924	French 589,506
November 26, 1924	Canad. 253,319
November 29, 1924	U. S. 1,583,681
April 24, 1925	Brit. 239,483
June 25, 1925	Brit. 247,140
August 31, 1925	U. S. 1,724,626
February 10, 1926	Dutch 21,841
October 5, 1926	Swiss, 124,290
November 24, 1926	Brit. 280,256
May 18, 1927	U. S. 1,786,125
October 3, 1927	U. S. 1,803,870
October 19, 1927	U. S. 1,940,646
November 7, 1927	U. S. 1,854,948
February 10, 1928	U. S. 1,745,907
February 20, 1928	Brit. 285,797
July 17, 1928	Brit. 300,606
August 26, 1928	Austral. 15,327/28
October 31, 1928	U. S. 1,934,057
February 25, 1929	Reissue 18,667 (U. S.)
March 9, 1929	U. S. 1,820,394
March 12, 1929	Canad. 293,233
June 6, 1929	U. S. 1,967,024
October 25, 1929	Canad. 333,642
February 15, 1930	Brit. 350,897

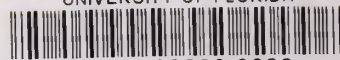
<u>Filing date</u>	<u>Patent</u>
March 11, 1930	U. S. 1,847,540
March 12, 1930	U. S. 2,006,456
August 23, 1930	U. S. 1,928,256
August 23, 1930	U. S. 1,928,968
September 10, 1930	Brit. 362,526
November 28, 1930	Ger. 555,117
March 12, 1931	U. S. 2,013,028
March 12, 1931	U. S. 2,042,296
May 4, 1931	U. S. 2,011,428
July 5, 1931	Ger. 564,018
August 20, 1931	U. S. 1,938,652
October 23, 1931	Canad. 338,896
November 27, 1931	Brit. 377,069
December 12, 1931	Erit. 378,300
December 12, 1931	Canad. 345,383
December 24, 1931	Canad. 340,315
January 4, 1932	Brit. 394,977
January 21, 1932	U. S. 1,940,899
February 7, 1932	Ger. 643,804
April 1, 1932	Canad. 338,897
May 14, 1932	U. S. 2,035,546
July 8, 1932	U. S. 1,954,517
September 8, 1932	U. S. 2,087,599
September 12, 1932	U. S. 2,058,200
October 19, 1932	U. S. 2,006,227
October 29, 1932	U. S. 2,066,184
November 22, 1932	Ger. 630,483
February 20, 1933	U. S. 1,942,104
March 17, 1933	U. S. 2,058,832
March 27, 1933	U. S. 1,942,827
April 10, 1933	U. S. 1,945,312
May 23, 1933	U. S. 1,963,838
October 2, 1933	U. S. 2,043,267
October 21, 1933	U. S. 2,096,885
November 15, 1933	U. S. 2,090,109
December 12, 1933	U. S. 2,024,392
January 24, 1934	Canad. 348,379
January 24, 1934	Canad. 348,380
February 28, 1934	French 769,445
March 16, 1934	Swedish 86,083
March 16, 1934	Swed. 87,625
March 19, 1934	Brit. 434,660
April 4, 1934	U. S. 2,083,098
April 28, 1934	U. S. 2,052,374
May 31, 1934	U. S. 2,104,757
June 5, 1934	U. S. 2,030,584
September 24, 1934	French 778,844
October 29, 1934	Brit. 446,576

Filing Date

Patent

December 17, 1934	Canad. 357,959
January 11, 1935	Austral. 20,894/35
January 11, 1935	Brit. 432,626
March 20, 1935	U. S. 2,049,725
March 30, 1935	Brit. 437,171
August 27, 1935	French 794,206
August 29, 1935	Canad. 362,903
September 4, 1935	U. S. 2,056,438
September 11, 1935	Ger. 646,570
October 25, 1935	U. S. 2,096,678
October 28, 1935	Canad. 369,499
October 29, 1935	French 797,052
January 3, 1936	U. S. 2,070,603
March 7, 1936	U. S. 2,089,766
December 24, 1936	Canad. 369,390
July 3, 1937	U. S. 2,103,195

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